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Polyethylene

New Developments and Applications

Edited by Arpit Sand and Jaya Tuteja

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Contributors
Jaya Tuteja, Aparna Vyas, Arpit Sand, Gurjeet Singh, Neeraj Agarwal, Sonia Saleem, Mohamed Abdel Maksoud, Ahmet Hakan Yilmaz, Bülend Ortaç, Saliha Mutlu, Sevil Savaskan Yilmaz, Normurodov Nurbek Fayzullo Ugli, Ashurov Nigmat Rustamovich, Ramez Labib, Ripal Amin, Lisa Hoffman

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Meet the editors

Dr. Arpit Sand is currently an associate professor in the Department of Chemistry, Manav Rachna University, Faridabad, India. He received his BSc in Science and MSc in Chemistry from the University of Allahabad, India, in 2004 and 2006, respectively. He received his Ph.D. in Chemistry from the same university in 2010. Dr. Sand is an editorial board member for *Polymer Synthesis* and *Journal of Polymer Science*. He is an academic book editor and a reviewer for international journals including *Carbohydrate Polymers*, *International Journal of Biological Macromolecules*, and *Fibers and Polymers*, among others. Dr. Sand's previous roles include assistant professor (guest faculty) in the Department of Chemistry, University of Allahabad; research associate at the National Physical Laboratory (NPL) New Delhi; postdoctoral fellow at Gwangju Institute of Science & Technology (GIST), Korea; postdoctoral fellow at Karolinska Institutet, Sweden; junior researcher at Brno University of Technology, Czech Republic; and researcher at Soongsil University, South Korea. He has more than 10 years of teaching and research experience. He is also a life member of the Indian Science Congress and Green Chemistry Network center. He has made significant contributions to the modification and characterization of graft copolymers and films decorated with chalcogenide quantum dots to tune the energy band gap for solar energy harvesting applications. His research interests include polymer synthesis using different polymerization techniques. He has authored more than twenty-four international research and review articles in reputed SCI journals.

Dr. Jaya Tuteja is an assistant professor in the School of Basic Science, Galgotias University, India. She earned her Ph.D. in Materials Science from the Japan Advanced Institute of Science and Technology (JAIST) in 2015, where she was recognized as an "Excellent Doctorate Student." She has published eight research papers in reputed international journals, eight book chapters, and two books. Her research articles have garnered many citations and one award. She has also filed two patents. Before joining Galgotias University, Dr. Tuteja worked at Manav Rachna University, India. She also has three years of industry experience as a chromatography specialist. Her research areas include the development, characterization, and application of heterogeneous catalysts for effective conversion of biomass to value-added products, nanoparticle synthesis and nanoparticle-based heterogeneous catalysts, bimetallic nanoparticles, carbohydrate chemistry, and analytical techniques for compound identification.

Contents

Preface

Dear Reader,

Welcome to the forefront of innovation in the realm of polyethylene. In this volume, we delve into the dynamic world of polyethylene, exploring its new developments, burgeoning applications, and the transformative impact it continues to exert across industries worldwide.

Polyethylene, with its versatile properties and myriad applications, stands as a cornerstone of modern materials science. From its humble beginnings to its current status as one of the most widely used polymers globally, the journey of polyethylene is a testament to human ingenuity and technological advancement.

In this book, we are privileged to present the collective insights and expertise of leading researchers, engineers, and practitioners in the field of polyethylene. Dr. Arpit Sand and Dr. Jaya Tuteja, distinguished experts in polymer chemistry and materials engineering, have meticulously curated a comprehensive exploration of the latest developments and innovative applications of polyethylene.

From novel synthesis techniques to cutting-edge applications in sectors ranging from packaging and construction to health care and beyond, this volume offers a panoramic view of the evolving landscape of polyethylene. Each chapter represents a collaborative endeavor, combining theoretical rigor with practical relevance to provide readers with a nuanced understanding of this remarkable polymer.

As we embark on this intellectual odyssey, we invite you, the reader, to join us in uncovering the boundless potential of polyethylene and its role in shaping the future of materials science and technology.

Sincerely,

Arpit Sand
School of Sciences,
Manav Rachna University Faridabad,
Haryana, India

Jaya Tuteja
School of Basic Sciences,
Galgotias University Greater Noida,
Uttar Pradesh, India

Chapter 1

Introductory Chapter: Polyethylene – Its Properties and Application in Industry and in Households

Jaya Tuteja, Aparna Vyas and Arpit Sand

1. Introduction

1.1 History of polyethylene

It is interesting to know that the most utility item of modern day was discovered accidentally approximately 90 years ago. In 1933, Reginald Gibson and Eric Fawcett at the Imperial Chemical Industries (ICI) in the UK were trying to react ethylene with benzaldehyde under high pressure but ended up with a white waxy substance instead [1]. The Scientist discovered that the compound on heating was not exploding, which was quite surprising. He further heated it and let it cool down overnight and further noticed that the compound had turned into a white paste, which later on was known as "Polytheylene." The term "plastic" is given to polyethylene (PE), as the plastic signifies the ability of a substance to mold itself into a variety of shapes. Its unique ability to get into different shapes is attributed to its composition that possesses C, N, Si, H, O, and N.

In the early 1940s, researchers at ICI and later at DuPont in the United States of America continued to develop polyethylene [2]. They realized that the process required high pressure and worked on improving production methods. In 1939, Paul Hogan and Robert Banks at ICI developed the first practical method for producing polyethylene by using a high-pressure process. This marked the beginning of large-scale production [3].

1.2 Applications of polyethylene

From there till date, this polyethylene and plastic have changed our life to a great extent. Polyethylene is widely used in packaging materials, including plastic bags, plastic films, and containers. In the medical field, polyethylene is used to manufacture various devices such as syringes, catheters, joint implants, and medical tubing [4]. Its biocompatibility and resistance to chemicals make it a valuable material for these applications [5]. Polyethylene is used in the construction industry for pipes, sheets, and insulation materials. Its durability, resistance to corrosion, and flexibility make it a popular choice for water and gas distribution systems. Many everyday items, such as toys, household items, and kitchenware, are made from polyethylene.

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Figure 1.
Uses of plastics in various applications.

Its versatility allows for a wide range of shapes and sizes. In automobiles, polyethylene is used for various components, including bumpers, fuel tanks, interior trims, and electrical insulation. It provides lightweight, impact-resistant solutions. Agricultural films made from polyethylene are used to cover crops, protecting them from pests, weeds, and adverse weather conditions. Polyethylene fibers are used in the production of a variety of textiles, including ropes, nets, and fabrics. Polyethylene is used in the production of various personal care items such as disposable diapers, sanitary napkins, and medical gloves. Overall, polyethylene's versatility, durability, and cost-effectiveness have made it a fundamental material in modern society, influencing a wide range of industries and aspects of daily life (**Figure 1**).

2. Structure, characteristics, and properties of polyethylene

Polyethylene is a polymer made up of repeating units of ethylene. It is a member of the polyolefin family of polymers. The polyethylene polymers are formed by condensation reaction, which was first produced by the scientist Von Pechmann in 1898 from the ether solution of diazomethane [6]. A similar product was observed by the scientist Bamberger in 1900 [7]. Just by observing the chemical structure of polyethylene, it seems quite simple. It includes the repetition of the same unit methylene along with some inclusion of comonomer at different places. This simple looking structure exhibits diversity, the largest diversity in the products formed by them.

There are three important or we can say fundamental molecular forces that contribute to its versatility and they are (1) the molecular weight and its distribution, (2) amount of comonomer and its distribution, and (3) short-chain/long-chain branching of monomers. Considering these factors, there are a variety of PEs available in the market. They are classified as:

2.1 High-density polyethylene (HDPE)

It is among the mostly used thermoplastic, as it is available in plenty, is pocket friendly, and possesses superior properties [8]. HDPE is known for its high strength-to-density ratio and is widely used in a variety of applications due to its versatile properties. It is a type of thermoplastic polymer made from petroleum. It possesses lesser branching, thus having stronger forces and high tensile strength. The HDPE has found its industrial applications in toys, packaging, construction, etc. HDPE is popular because it is relatively inexpensive, chemically resistant, tough, and can be easily molded or extruded into various shapes. It also has a lower environmental impact compared to some other plastics, as it is recyclable and can be reused in a variety of applications.

2.2 Low-density polyethylene (LDPE)

LDPE stands for low-density polyethylene. It is a type of thermoplastic polymer that is commonly used in a wide range of applications. LDPE is known for its flexibility, transparency, and resistance to moisture. It is commonly used in packaging materials such as plastic bags, shrink wraps, and containers. Additionally, LDPE is used in various consumer products like toys, squeeze bottles, and some types of medical devices.

Low-density polyethylene (LDPE) is created through the polymerization of ethylene, a basic hydrocarbon compound, under high pressure. This process produces a material with a lower density compared to other types of polyethylene, which gives LDPE its characteristic flexibility.

2.3 Linear low-density polyethylene (LLDPE)

LLDPE is a type of thermoplastic polymer made from the monomer ethylene. It is called “linear” because it has a more linear structure compared to other forms of polyethylene, which gives it certain unique properties. LLDPE is known for its flexibility, toughness, and relatively low density. It is commonly used in applications where a combination of these properties is desired.

2.4 Ultra-low density polyethylene (ULDPE)

ULDPE is a type of thermoplastic material known for its exceptionally low density compared to other forms of polyethylene. This characteristic makes ULDPE particularly suitable for applications where lightweight and flexible materials are required.

3. Conclusion

In conclusion, polyethylene (PE) stands as a remarkable material with a wide range of applications both in industry and households, owing to its exceptional properties. Its versatility, durability, and cost-effectiveness make it a cornerstone of modern manufacturing and consumer goods.

In industry, PE’s high chemical resistance and excellent electrical insulating properties make it a preferred material for various applications. It serves as a fundamental component in pipelines, cable insulation, and a variety of packaging materials. Its ability to be easily molded and extruded allows for intricate designs and customized solutions in a multitude of sectors.

Within households, PE plays an integral role in everyday life. From packaging materials like bags and containers to essential items like bottles, it provides convenience and protection for a wide array of goods. Its use in insulation materials, flooring, and furniture underscores its importance in enhancing the comfort and functionality of living spaces. Furthermore, in medical and personal care applications, PE ensures safety and hygiene.

However, it is crucial to recognize the environmental challenges associated with PE production and disposal. As a petroleum-based product, its production contributes to resource consumption and pollution. Proper recycling and responsible disposal practices are imperative to mitigate its impact on the environment.

In light of these considerations, ongoing efforts to develop sustainable alternatives and improve recycling technologies are essential. Despite its widespread use, the responsible use and management of polyethylene remain paramount in achieving a more sustainable and environmentally conscious future.

Author details

Jaya Tuteja[1], Aparna Vyas[2] and Arpit Sand[2]*

1 School of Basic Sciences, Galgotias University Greater Noida, Uttar Pradesh, India

2 School of Sciences, Manav Rachna University Faridabad, Haryana, India

*Address all correspondence to: arpit@mru.edu.in

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Chapter 2

Degradation Pathways and Ecological Consequences of Use of Polythene

Gurjeet Singh and Neeraj Agarwal

Abstract

Due to its adaptability and affordability, polyethylene, a synthetic polymer that is often utilized, has made a substantial contribution to modern civilization. However, due to its widespread usage, there is concern about its environmental persistence and potential ecological effects. This article seeks to present a thorough explanation of the mechanisms involved in polyethylene degradation, the environmental repercussions of its buildup, and proposed remediation techniques to lessen those effects. The study examines the fundamental processes of several degradation routes, such as biological degradation etc.. Efforts to address the ecological consequences of polythene use include reducing plastic waste management, developing biodegradation products.

Keywords: polythene, degradation, microbial enzymes, plastic pollution, environmental impact

1. Introduction

The five most common petroleum-based polymers used to make single-use plastic products are polyethylene terephthalate (PET), polypropylene (PP), high density polyethylene (HDPE), polyvinyl chloride (PVC), and low density polyethylene (LDPE). The most prevalent petroleum-polymer on earth, LDPE, is responsible for up to 64% of single-use plastics that are discarded shortly after use, causing a massive and fast buildup in the environment [1, 2]. The negative impacts of basically "non-biodegradable" LDPE rubbish buildup in landfills and seas are growing despite recycling and energy recovery measures. Micro-plastics may now be found everywhere on the planet, including in the arctic snow, according to mounting evidence [3]. Finding an environmentally appropriate disposal method is thus required [4]. Contrary to biological waste that is dumped, polyethylene (PE) and other petroleum-based polymers are particularly resistant to natural biodegradation processes. The scientific literature has several studies on the biodegradation of synthetic polymers, including PE in particular. Thirteen evaluations of the microbes involved in the physical and microbial biodegradation processes have been published since 2008.

Although microbial breakdown of PE has been observed in various studies, significant degradation of PE wastes at usable sizes has not yet been achieved. We have been limited in our ability to develop a biochemically based knowledge of the mechanisms and processes involved in PE degradation due to the lack of a concrete definition of polyethylene biodegradation that may lead to testable hypotheses. Early investigations on microbial biodegradation attempted to demonstrate how microbial activity may change the tensile strength, water absorption, and crystallinity of plastics [5].

Pirt (1980) [6] conducted the first investigation of the microbial biodegradation of polymers. Ten years later, 0.2% less PE weight was present, according to Albertsson and Karlsson [1]. Otake et al. [7] found that PE polymers exhibited surface alterations after being buried in soil for 10 to 32 years. It was discovered that LDPE thin film deterioration was relatively high. Despite the fact that parts of the PE films with considerable deterioration were characterized by whitening with tiny holes, the overall rate of degradation was still fairly low even after years of contact with soil microorganisms.

Some researchers have investigated the aerobic biodegradation of treated polyethylene and/or polyethylene modified by the addition of additives ("addivitated") PE in simulated soil burial and mature compost [3, 8], as well as in natural aquatic environments under laboratory conditions [9, 10]. Living microbial consortia are present in several kinds of soil [11]. Others looked at the microorganisms that cause LDPE to biodegrade in soil [12]. The biodegradability of thermally and photochemically damaged addiviated LDPE films by microorganisms adsorbed on the surface of PE films buried in agricultural soil was assessed in a research by Abrusci et al. [13] whiteness with tiny holes that defines it.

Typically, as part of microbial degradation test investigations, microorganisms from diverse sources are isolated to ascertain the optimal microbial power to degrade polymeric PE chains. Researchers have isolated potential microorganisms from a range of soil types, including garden soil, forest soil, waste soil, mangrove soil, and soil covered in agricultural PE films for soil mulching [14–17]. Alternative sources for the isolation of high potential microorganisms that deteriorate PE included landfills, solid waste dumps, and plastic garbage (municipal solid soil) [4, 18–23], water [2], waste water or sewage sludge, oil-contaminated soil, and even waxworm larvae [23].

Numerous bacteria from a small number of text were found to be present in these trials; however, not all of them were involved in the breakdown of PE (**Table 1**). Following the bacteria's initial isolation, the capacity of each isolate to use treated and/or untreated polyethylene was examined in isolated shake-flask cultures throughout a range of time periods. The majority of these bacteria were identified using the sequencing of 16S ribosomal RNA genes following PCR amplification. The third phase was estimating biodegradation using PE-degrading bacteria on polyethylene particles or films using various approaches.

Comparisons of the various biodegradation results are not significant due to the large diversity of PE materials employed and the vast range of growth conditions. This emphasizes the requirement for standardized approaches and procedures to comprehensively investigate the biodegradation of synthetic polymers. We need to identify the differences between degradation and deterioration as well as what the biodegradation process entails in order to resolve any difficulties brought up by stories of attempts at microbial biodegradation of PE that failed. The conditions that promote the microbial destruction of PE are discussed in the sections that follow, along with how these factors led to reports of incorrect PE biodegradation percentages. Then, we provide an appropriate explanation of the biodegradation process that will make it possible to interpret the findings of biodegradation in an accurate manner.

Genus (and Species)	**Source**	**Biodegradation result**
Acinetobacter bumannii [24]	Municipal landfill	Biomass production
Arthobacter defluvii [25]	Dumped soil area	20–30% W.L.
Bacillus amyloliquefaciens [26]		
Bacillussubtilis [27]		
Bacillus pumilus [28]	Pelagic waters	1.5%–1.75% W.L.
Bacillus subtillis [29]		
Bacillus ssp. [30]	Waste coal, a forest and an extinct volcano crater	Reduction of mechanical properties by 98%
		No W.L. detected
Bacillus sphericus [31]	Shallow waters of ocean	3.5% and 10%
		9% and 19%
Bacillus megaterium [7]	Soil	7–10% mineralization
Bacillus subtilis [32]		
Bacillus cereus (MIX together) [33]		
Bacillus amyloliquefaciens [34]	Solid waste dumped	11–16%
Bacillus subtilis [34]	MCC No. 2183	9.26% W.L.
Bacillus pumilus M27 [35]	Pelagic waters	1.5–1.75 W.L. %
Bacillus subtilis H1584 [35]		
Brevibacillus borstelensis [36]	DSMZ	17% W.L.
Brevibacillus [37]	Waste disposal site	37.5% W.L.
Chryseobacterium gleum [37]	Waste water activated sludge soil	—
Comamonas sp. [38]	Plastic debris in soil	Changing in chemical properties
Delftia sp. [39]	Plastic debris in soil	Changing in chemical properties
Kocuria palustris M16 [40]	Pelagic waters	1%
Microbacterium paraoxydans [18]	Having Gene bank ID	61% W.L.
Pseudomonas sp. [19]	Mangrove soil	20.54% W.L.
Pseudomonas aeroginosa [20]	Petroleum contaminated beach soil	40.8% W.L.
Pseudomonas sp. [20]	Beach soil contaminated with crude oil	4.9%–28.6% CO2 production
Pseudomonas sp. [21]	Garbage soil	37.09% W.L.
Pseudomonas citronellolis [22]	Municipal Landfill	17.8% W.L.
Pseudomonas sp. [23]	Having Gene bank ID	50.5% W.L.
Pseudomonas aeroginosa [36]	ATCC	9–20%
Pseudomonas putida [37]		
Pseudomonas siringae [38]		

Genus (and Species)	Source	Biodegradation result
Pseudomonas sp. [4]	Waste disposal site	40.5% W.L.
Rhodococcus ruber [1]	PE agricultural waste in soil	Up to 8% W.L.
Rhodococcus ruber [2]	PE agricultural waste in soil	0.86% W.L./week
Rhodococcus ruber [2]	PE agricultural waste in soil	1.5%–2.5% W.L.
		Reduction of 20%.in Mw and 15%. in Mn
Rhodococcus rhorocuros [41]	ATCC	60% mineralization
Rhodococcus rhorocuros [42]	ATCC 29672	Different amount of mineralization
Rhodococcus sp. [5]	Waste disposal site	33% W.L.
Rhodococcus sp. [6]	Three forest soil	Confirmation of Adhering
Staphylococcus arlettae [3]	Various soil environments	13.6% W.L.
Stentrophomonas sp. [4]	Plastic debris in soil	Changing in chemical properties
Stentrophomonas pavanii [8]	Solid waste dump site	Confirmed by FTIR
Streptomyces spp. [9]	Nile River Delta	3 species showed slight W.L.

Table 1.
Bacteria employed in research on the biodegradation of polyethylene (PE).

2. Biological degradation of PE

There are four steps to the whole biodegradation process: biodeterioration, biofragmentation, bioassimilation, and mineralization. However, access sites in the PE structure are necessary for microorganisms to start fragmenting before they can start attacking PE. As a result, before the presence of microorganisms, oxidation of PE polymers happens by abiotic processes such as ultraviolet (UV) radiation exposure combined with heat and/or environmental chemicals. It is well known that thermal aging frequently occurs in conjunction with PE oxidation, particularly UV-induced PE oxidation. The mechanisms of polymer change have also been well shown. According to earlier studies, when PE is exposed to UV radiation or oxidizing agents, carbonyl groups are produced in the alkane chains. These carbonyl groups are then further hydrolyzed by microorganisms, which catabolize the shorter PE chain reaction products (fragmentation). In this method, the polymer chain initially absorbs UV light, which causes radical production. At some point, oxygen is taken in, hydroperoxides are created, and carbonyl groups are created (**Figure 1**). The carbonyl groups proceed through Norrish Type I and/or Type II degradation with additional UV exposure. Additionally, pro-oxidants or contaminants might start photo-oxidation. Additionally, UV-degradation might start at spots where minute amounts of ketone or hydroperoxide groups were added during fabrication or production.

Infrared spectroscopy (IR) measurements of the degree of carbonyl group adsorption can be used to monitor the oxidative degradation of polyolefins. The concentration levels of carbonyl compounds determined by ATR-FTIR were often represented as a carbonyl index (C.I.), which is defined as the ratio of carbonyl and methylene absorbances. The ratio of the methylene absorption band at 1435 cm1 (the CH2 scissoring peak) to the carbonyl peak at 1714 cm1 taken as an internal thickness band

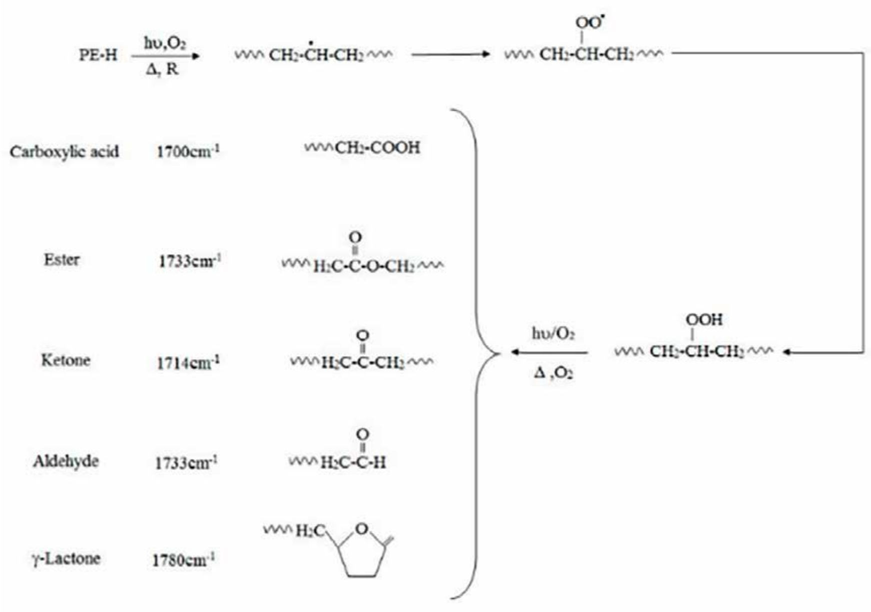

Figure 1.
Degradation pathways of polyethylene containing pro-oxidant additives.

(CI = A1714/A1435). Even after storage in an abiotic environment, photo-oxidation and increased stress both accelerate the production of carbonyl groups.

3. Biodeterioration of PE

Some microbes can start the oxidation process on their own, via the process of "hydroperoxidation," in addition to the abiotic degradation of PE materials. "Biodeterioration" is the word used to describe this. However, it is currently unclear whether PE that has been oxidized in this way can eventually be broken down by microbes [9]. Different pro-oxidation additives (prodegradants) have been added to the structure of polyethylene products to make them "oxo-degradable" in various investigations of the microbial breakdown of PE. "Addiviated" polymers are PE polymers that include additives that make them oxo-degradable. Materials used to make addiviated PE polymers oxo-degradable include polyunsaturated compounds, transition metals like iron, cobalt, manganese, and calcium, totally degradable plastic additives (TDPA) with different commercial names [7, 30, 31, 40], natural polymers (e.g., starch, cellulose, or chitosan), food grade dyes, or synthetic polymers containing ester, hydroxyl or ether groups [33, 35, 40] that are prone to hydrolytic cleavage by microorganisms. Abiotic factors like sunlight, heat, or both, as well as the addition of oxidizing chemical agents like nitric acid are used in some comparative studies of the microbial degradation of PE to start the degradation of raw and addiviated PE polymers and make the plastic more susceptible to microbial degradation. Following this, the impacts of various treatments on PE structure and microbial degradation were examined and compared to samples that had not been processed. The development of oxidized oligomers and alteration of the polymer are caused by a change in the fundamental structure of PE during the degrading process. The PE becomes brittle and vulnerable to additional oxidation by enzymes released by the microorganisms as a result of deterioration caused by physical, biological, or chemical factors. While PE's molecular structure is changing at this point, the polymer is not fragmenting or losing structure. An increase in entry locations for enzymes released by microbes and a decline in the polymer's mechanical or other physical qualities are two main characteristics of the degradation phase overall.

4. Experiments on microbial degradation of PE: contributing factors

The outcome and findings of PE biodegradation tests are significantly influenced by a variety of parameters in the microbial breakdown of PE polymers. Unfortunately, while planning and designing the trials that were described in the literature, these considerations were frequently ignored. As a result, the information provided in these papers about PE biodegradation has been inconsistent and inconclusive. Following is a description of these elements.

4.1 Polyethylene shape and structure

The ability of the microorganisms' secreted enzymes to reach the PE carbon chain is crucial for microbial breakdown. All PE materials have a simple linear carbon chain microstructure that is joined by hydrogen bonds. But polyethylene polymers can have a variety of densities and three-dimensional (3-D) structures (**Figure 2**), including low molecular weight polyethylene (LMWPE), linear low-density polyethylene (LLDPE), low-density polyethylene (LDPE), and high-density polyethylene (HDPE), depending on the manufacturing processes used.

PE often has a semi-crystalline structure as well. LDPE crystallinity ranges from 45–65%, depending on the type of processing. Short branches (10–30 CH3 groups per 1000 C-atoms) made comprised of one or more co-monomers like 1-butene, 1-hexene, and 1-octene are typically found in amorphous regions of LDPE. The LDPE chains near the surface are made more accessible by the branching system's prevention

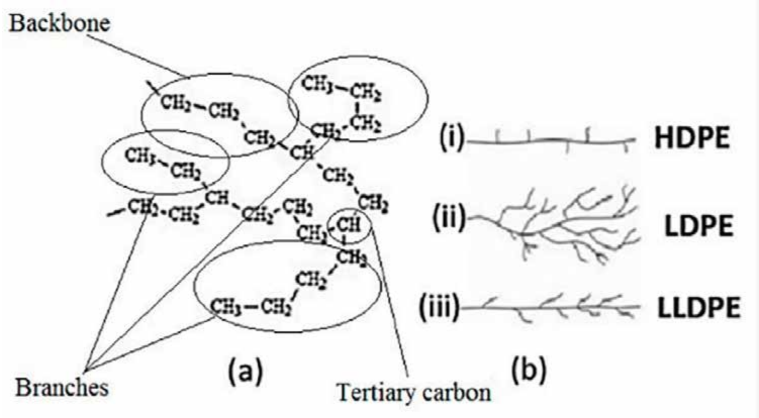

Figure 2.
Polyethylene structure.

of the PE molecules from stacking closely together, and the tertiary carbon atoms at the branch sites are left more vulnerable to assault. Additionally, amorphous areas are more likely to contain contaminants.

So that it is feasible to determine how much polymer is present, it is crucial that the structure and percentage of amorphous and crystalline areas in the polymer be recorded.

4.2 Modification of polyethylene

PE often has a semi-crystalline structure as well. LDPE crystallinity ranges from 45–65%, depending on the type of processing. Short branches (10–30 CH3 groups per 1000 C-atoms) made comprised of one or more co-monomers like 1-butene, 1-hexene, and 1-octene are typically found in amorphous regions of LDPE. The LDPE chains near the surface are made more accessible by the branching system's prevention of the PE molecules from stacking closely together, and the tertiary carbon atoms at the branch sites are left more vulnerable to assault. Additionally, amorphous areas are more likely to contain contaminants. So that it is feasible to determine how much polymer is present, it is crucial that the structure and percentage of amorphous and crystalline areas in the polymer be recorded.

But the primary goal of LDPE modification is to cause the polyethylene structure to deteriorate, allowing more access to the enzymes released by microorganisms during the biodegradation stage. Treatments alter the structure of PE, and as a result, investigations using various forms of PE with varying Mw, Mn, and/or molecular distributions have produced varying biodegradation outcomes. To effectively quantify microbial degradation and ascertain the only impact of microorganisms' activities, these changes in the biodegradation process need to be identified and documented.

4.3 Partial biodegradation versus complete degradation

The consumption and mineralization of whole, unaltered polymers, including the polymer's backbone, might be considered complete biodegradation of PE polymers. Microbes that can totally breakdown and mineralize virgin polyethylene have not yet been discovered, according to Yoon et al. Even so, it's possible to classify the numerous cases of PE biodegradation in the literature as incomplete biodegradation. As previously mentioned, PE polymers are composed of a complex of linear carbon chains held together by van-der-Waals interactions, accessible short side-chains with tertiary carbon that contain amorphous sections, terminal methyl-groups at the ends of chains, short branches, and small oxidative products, as well as numerous linear and branched n-alkane side-chains. Because the side-chains of PE mimic linear n-alkanes, they may serve as the first site of contact for bacterial enzymes that cause the polymers to partially degrade. Without fragmenting the polymer's backbone, low molar mass molecules and/or amorphous segments are removed from its surface. In contrast to the fragmentation of the backbone or pure PE polymers, weight loss during the early stages of PE degradation may be explained by the enzymatic hydrolysis of these readily accessible side chains. It is insufficient to conclude that polyethylene has completely degraded by looking at the development of microbes on agar plates containing the material. This has been one of the main issues with biodegradation experiments since it is necessary to establish complete biodegradation.

4.4 Other carbon sources' influence on biodegradation

There are various carbon sources that, in biodegradation tests, are frequently absorbed by bacteria during the initial phases of microbial breakdown and may interfere with the only carbon supply of PE. Establishing a growth curve for the bacteria under research using PE as the carbon source is advised as a solution to the issue. Changes in the development curve might signify the use of various carbon sources with varying degrees of accessibility to microbes [18]. Impurities that are integrated into PE chains or that adhere to the PE surface may include substances that bacteria can use as a source of carbon. Consumption of these contaminants can compete with or obstruct the use of PE as a carbon source. Incubating non-PE degrading bacteria, such as *E. coli*, along with the contaminated PE samples is one strategy to mitigate this issue. The contaminants would be consumed by the *E. coli* without changing the structure of the PE. After a certain amount of time, the pure PE may be removed, cleaned, and cultured with various microbes to see how well they degrade PE.

Generally speaking, two distinct groups of researchers have carried out experiments for the microbial breakdown of PE. Environmentalists in general are the groups of researchers who have studied the degradation of bulk PE materials of various types (LMWPE, LLDPE, LDPE, or HDPE) in natural settings such as soil, compost, or aquatic systems with mixed, undefined populations of microorganisms, without paying attention to microbial type. Any change seen is referred to as "biodegradation" whether it relates to appearance, weight loss, or mechanical qualities of the PE. The mechanisms influencing changes in the PE are unclear, and this strategy is mostly based on "trial and error." The distinction between deterioration and partial degradation is misunderstood by the authors of these works. On the other hand, these tests have the benefit of being carried out in the real world under actual environmental circumstances, and the outcomes accurately represent the deterioration of PE. The final conversion of PE to CO2 and biomass (mineralization via genuine biodegradation), is a topic of interest to microbiologists who have also studied PE breakdown. The biodegradation tests are carried out with specific species of microorganisms isolated using specialized medium from collections. In general, the tests' many components are clearly specified, and the authors are aware of how biodegradation works. The use of molecular biology and genomic sciences has started to pinpoint the precise genes and gene products involved in the breakdown of polyethylene in this regard. PE biodegradation is a complicated process that is impacted by a wide range of variables. Before being subjected to microbial treatment, a PE polymer chain may be exposed to various manufacturing, treatment, and sample preparation operations. The biodegradation process is complicated and uncertain since it involves a vast variety of bacteria with diverse behaviors and released chemicals. However, studies of polyethylene biodegradation experiments may be conducted from both chemical and microbiological perspectives.

5. Conclusion

The four steps of PE's biodegradation process are biodeterioration, biofragmentation, bioassimilation, and mineralization. Complete biodegradation of PE necessitates a decrease in the polymer's molar mass and molecular mass number as a result of fragmentation into smaller molecules that are then metabolized by microorganisms. However, the majority of investigations on the purported biodegradation of PE by microorganisms show biodeterioration and just a small number report biofragmentation. Furthermore, there is not enough proof to support bioassimilation and mineralization. Understanding the molecular processes of polyethylene biodegradation may be improved by investigating the genes and gene products that oxidize the alkane chains of polyethylene.

Author details

Gurjeet Singh* and Neeraj Agarwal
Department of Mechanical Engineering, IES College of Technology, Bhopal, M.P., India

*Address all correspondence to: gurjeet.singh@iesbpl.ac.in

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Chapter 3

Perspective Chapter: Polyethylene Glycol Use in Colonoscopy Preparation versus Chronic Constipation

Sonia Saleem

Abstract

Polyethylene glycol is a product with many pharmaceutical and industrial uses due to its hydrophilic nature. As a medication, it is part of a laxative class of drugs which is minimally absorbed in gastrointestinal tract increasing osmotic pressure resulting in softening of stools and increased bowel movements. There are certain FDA and non FDA-approved indications for its use. Its main use is for the treatment of constipation, fecal impaction, chronic constipation, and colonoscopy preparation. Its administration is elaborated according to different age groups and indications. Polyethylene glycol is over-the-counter medication as there is easy access to this drug, but as a health care provider, we need to emphasize the directions for its appropriate use with proper counseling regarding the onset of action as well as when to discontinue it which will yield improved patient results while minimizing adverse events.

Keywords: polyethylene glycol (PEG), constipation, colonoscopy preparation, laxative, awareness for its use

1. Introduction

Polyethylene glycol is a polyether compound mainly derived from petroleum. They are synthesized by polymerization of polyethylene oxide. These are available over a wide range of products with many applications including biomedical, pharmaceutical, industrial, chemical, commercial, and entertainment industry. Due to its hydrophilic nature, it is mainly utilized in cosmetic products, surfactants, emulsifiers, humectants, skin conditioners [1], and cleansing agents. It is used as an anti-foaming agent in foods and drinks [2]. It is used as an excipient in many pharmaceutical preparations in oral, parenteral, and topical forms [3]. The use of PEG hydrogels has been theorized by Ma et al. They proposed its use to address periodontitis (gum disease) by encapsulating stem cells in the gel which promotes healing in the gum [4]. PEGylated lipid is used as an excipient in both Pfizer and Moderna vaccines for SARS-CoV-2. Both vaccines contain mRNA encased in a bubble of molecules that is made up of polyethylene glycol. As of Dec 2020, there was a concern about polyethylene glycol causing hypersensitivity/allergic reactions [5], and also reported that two individuals recovered from anaphylactic shock [6, 7]. In Dec, US CDC stated in their jurisdiction that six cases of allergic reactions were recorded from over 250,000 vaccinations, and of those six, only one person had history of vaccination reaction [8].

The production of Polyethylene glycol was first reported in 1859. Both A. V. Lourenco and Charles Adolphe Wurtz independently isolated these products [9]. Polyethylene glycol is produced by the interaction of ethylene oxide with water, ethylene glycol oligomers, or ethylene glycol. The reaction is catalyzed by acidic or basic catalysts. Polyethylene glycol is a hydrophilic (water-loving) polymer that is made by reacting ethylene oxide with ethylene glycol (the simplest member of glycol family) or with ethylene glycol oligomers (molecules that contain repeating units) or water.

As a medication, it belongs to laxative class of drugs. It works by causing water to be retained in stool resulting in softening of stool as well as increasing the number of bowel movements.

It is used in the management and treatment of chronic constipation and fecal impaction with polyethylene glycol powder being mixed with liquid and then taken by mouth, As an osmotic laxative, the compound works by forming hydrogen bonds with water molecules which prevents the re-absorption of water in the gastrointestinal tract, causing water retention in the stool and increasing osmotic pressure. Water retention softens the stool making it easier to pass; the number of bowel movements also increases. Potential side effects include nausea, bloating, and intestinal cramping; more serious effects include diarrhea and in sensitive individuals, hives. It is also used for colonoscopy preparation as a way to cleanse the bowel to help provide a clear image. It is typically mixed with two liters of sports drink, the day before the procedure.

This chapter includes polyethylene glycol used in chronic constipation and for colonoscopy preparation, its potential side effects, contraindications, method of administration, and when and how to discontinue it in case of chronic constipation. It is over-the-counter medication. There is an easy access to this drug. The objective is to spread awareness among the patients and healthcare teams which will yield improved patient results while minimizing adverse effects that are still possible with over-the-counter medication (like PEG).

2. Indications

2.1 FDA-approved indications

- Treatment of constipation in patients 17 years or older.
    - It is preferred over other agents due to its limited side effects and better testing profile.
    - Polyethylene glycol laxative is safer than Polyethylene glycol electrolyte lavage Solution as there is no salt absorption.

2.2 Non-FDA approved indications

- Colonoscopy preparation
- Fecal impaction

- Chronic constipation
- Constipation in pediatric patients

2.3 Mechanism of action

Polyethylene glycol is hydrophilic, and minimally absorbed in the gastrointestinal tract as a medication. It forms hydrogen bonds with water molecules. For this reason, it can prevent the re-absorption of water which causes water retention in stool and increases the osmotic pressure. As a result, stool softens and bowel movements occur more frequently [10, 11].

3. Administration

- Polyethylene glycol-3350 comes as a powder to be mixed with liquid and taken by mouth.
- It is usually taken once a day as needed for up to 2 weeks.
- To use powder, follow these steps [11];
    1. If using polyethylene glycol from the bottle, use the measuring line on the bottle cap to measure a single dose (about one a heaping tablespoon).
    2. If using a polyethylene glycol packet, each packet contains a single dose.
    3. Pour the powder into the cup containing 08 ounces (about 240 ml) of water, juice, soda, coffee, or tea.
    4. Stir to dissolve the powder.
    5. Drink immediately
- It may be habit-forming. Do not take a larger dose, take it more often or take it for a longer period of time.
- It may take two to 4 days for polyethylene glycol-3350 to produce a bowel movement.
- In the adult population, polyethylene glycol should not be used for more than 2 weeks as an over-the-counter medication unless instructed by a physician. If needed more chronically, it can be taken for up to 6 months [12].
- It is also used for the preparation of colonoscopy. Polyethylene glycol is used to cleanse the bowel to help in providing a clear image of the mucosa.
- It is administered by mixing 238 g in 2 liters of sports drinks on the day before the procedure. Guidelines also recommend doing split dose when colonoscopy is in

the afternoon, administer one liter on the day before the procedure and one liter on the day of procedure.

4. Dietary instructions

- To treat constipation, along with using polyethylene glycol as a laxative, eat a well-balanced diet that includes fiber-rich foods such as unprocessed bran, whole grains, bread, fresh fruits, and vegetables.
- Drink plenty of fluid.
- Exercise regularly

5. Special patiena: Population

Pregnancy

- It is a pregnancy category C drug.
- The systemic absorption of polyethylene glycol is believed to be minimal, therefore it is less likely to cause any severe effect on neonate.

Breast feeding women

- No specific recommendation, as it has poor systemic absorption [13], so there should be minimal to no excretion in the breast milk.

Pediatrics

- For children more than 2 years of age, it can be used for chronic constipation and fecal impaction on a weight-based dosage adjustment.
    - For fecal impaction; 1–1.5 g/kg/day for 3–6 days with a maximum of 6 consecutive days.
    - For Chronic constipation; initial dose. 0.4 g/kg/day orally
- Maintenance dose; 0.2–0.8 g/kg/day adjusted to response
- The recommendation is to continue for at least 2 months and discontinue gradually once symptoms resolve for at least months [14].

Patients with hepatic impairment; No dose adjustment is required.
Patients with renal impairment; No dose adjustment required.
Geriatric patients; No dose adjustment is required.

6. Adverse effects

In adults
Common; Common side effects of oral administration of polyethylene glycol.

- Flatulence/Diarrhea
- Nausea
- Stomach cramps
- Rectal hemorrhage [15, 16]
- After using polyethylene glycol Electrolyte Lavage Solution following topical administration—Urticaria [12, 17]

Rare;

- Acidosis reported in literature and case reports with intravenous administration of drugs utilizing polyethylene glycol as a vehicle and repeated topical dermal exposure of polyethylene glycol in burn patients [18, 19].
- Proximal renal tubular necrosis with oliguria, azotemia, and renal failure following intravenous administration of polyethylene glycol and topical application in burn patients [20].

In children

- Seizures
- Tremor
- Obsessive-compulsive behaviors (including repetitive chewing and sucking)
- Paranoia/mood swings
- Headache
- Sedation/lethargy
- Aggression/anxiety

7. Contraindications

- Patient with known or suspected bowel obstruction, appendicitis, inflammatory bowel disease, perforated bowel, and hypersensitivity to polyethylene glycol or any component of formulation.
- Precautionary measures are necessary for patients with electrolyte imbalances and patients with renal impairment [21].

8. Monitoring

- Before initiating, rule out symptoms and signs of bowel obstruction.
- Patient education regarding proper use of PEG and monitoring for therapeutic efficacy including decreased abdominal discomfort, pain, and bowel movement in two to 4 days.
- Electrolyte imbalances should be monitored periodically with prolonged, frequent, or excessive use [21].
- Pediatric patients on long-term use of polyethylene glycol should also be monitored for neuropsychiatric symptoms.
- Patients should discontinue use of polyethylene glycol if they experience loose stools.
- Clinicians should taper down patients on chronic therapy.

9. Toxicity/overdose

- Due to limited exposure, toxicity to polyethylene glycol is very rare.
- Diarrhea leading to dehydration especially if water intake is minimal, can result in confusion and potentially seizures [22].
- Renal injury and metabolic acidosis.
- There is no antidote in case of polyethylene glycol overdose

10. Polyethylene glycol use in colonoscopy preparation

A colonoscopy is the principal diagnostic tool to inspect mucosa of the colon for any pathology such as inflammation, polyp, adenoma, or cancer. It is preferred over other imaging modalities like barium enema or computed tomography (CT) colonography in many situations because of its capacity to intervene and sample or remove pathology encountered.

The pre-requisite for optimal endoscopic visualization is adequate bowel cleansing as it affects safety, quality, efficacy, and diagnostic accuracy of the procedure. The colonoscopic detection rate of lesions is affected by bowel preparation quality [23]. Previous studies showed that at the time of colonoscopy, about 20% of patients had inadequate bowel preparation [24]. Inadequate bowel preparation may result in an increased risk of missing lesions, decreased cecal intubation rate, increased patient discomfort due to repeated washings and suctioning, prolonged procedure time, higher risk of complications, increased rate of the canceled procedure with increasing costs and need to repeat the procedure [25]. So ideally the colon cleansing method should be fast, safe, and able to do proper cleaning with minimal side effects and discomfort for the patient [26]. There are different agents used for colonoscopy preparation, including polyethylene glycol, sodium phosphate, pyrosulphate solution, and oral sulfate solution. There are two regimens used for colonoscopy preparation including split dose and day before cleansing method [27]. The agents which are used commonly include sodium phosphate and polyethylene glycol electrolyte solution. PEG and sodium phosphate, both are osmotic laxatives that cause water retention in stools and increase stool frequency. The preference for one preparation over the other is guided by consensus regarding two preparations, relative safety, tolerability, and effectiveness [28]. Preferentially PEG-based preparations are given in patients who have renal insufficiency. It is also preferred in patients who have diabetes mellitus or congestive cardiac failure [29].

10.1 Patient selection

Patient selection for colonoscopy focuses on the indication for the procedure and patient comorbidities to enable proper risk stratification.

The American Society of Gastrointestinal Endoscopy has outlined general principles for the appropriate use of endoscopy [30]. Endoscopy may be pertinent if any of the following criteria are fulfilled:

- There is an effective change in the patient's management after results
- There is failure of empiric treatment of a benign disease
- There is anticipation of therapeutic intervention
- It is used as an alternative to radiologic evaluation

On the other hand, when the results are not expected to change the management plan appropriately or just for the sake of benign disease follow-up that hase healed, there is no need to undergo a colonoscopy, except to do a surveillance colonoscopy for anticipated pre-malignant condition (**Figures 1**–**5**).

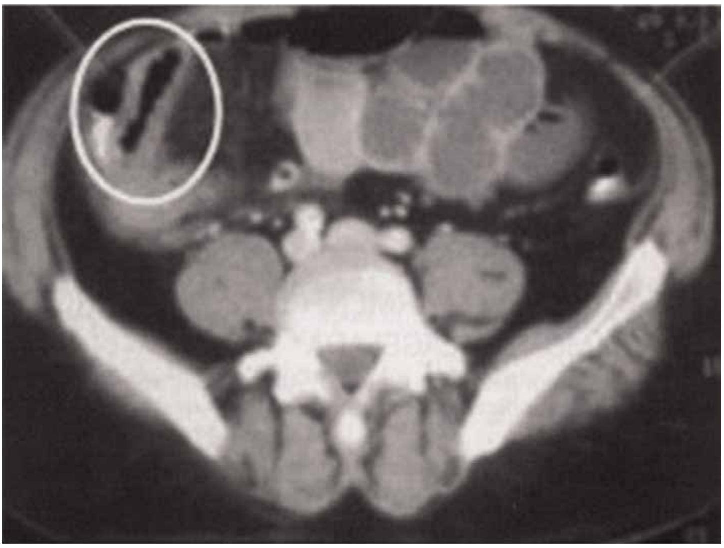

Figure 1.
Abdominal computed tomography scan showing thickening of the distal ileum.

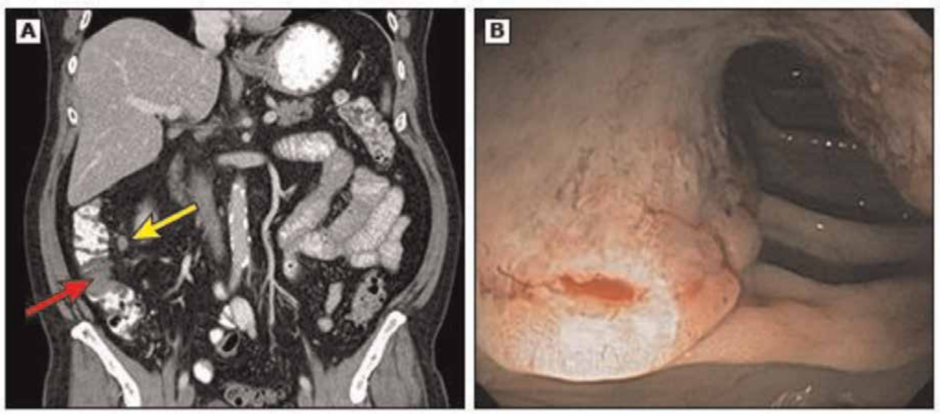

Figure 2.
Colon cancer is seen on CT scan and colonoscopy. (A) Computed tomographic (CT) scan showing a filling defect in the ascending colon (red arrow) along with an involved lymph node (yellow arrow). (B) Colon cancer identified in the ascending colon on subsequent colonoscopy.

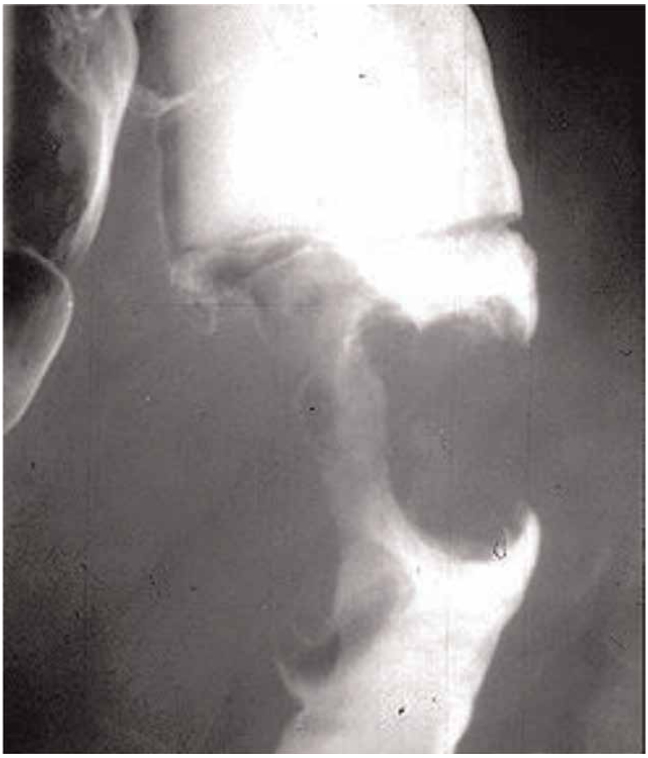

Figure 3.
Cancer of the colon as seen on barium enema. Double-contrast barium enema shows an apple-core lesion surrounding the lumen of the descending colon.

10.2 Indications; both diagnostic and therapeutic

- Lower GI Bleeding

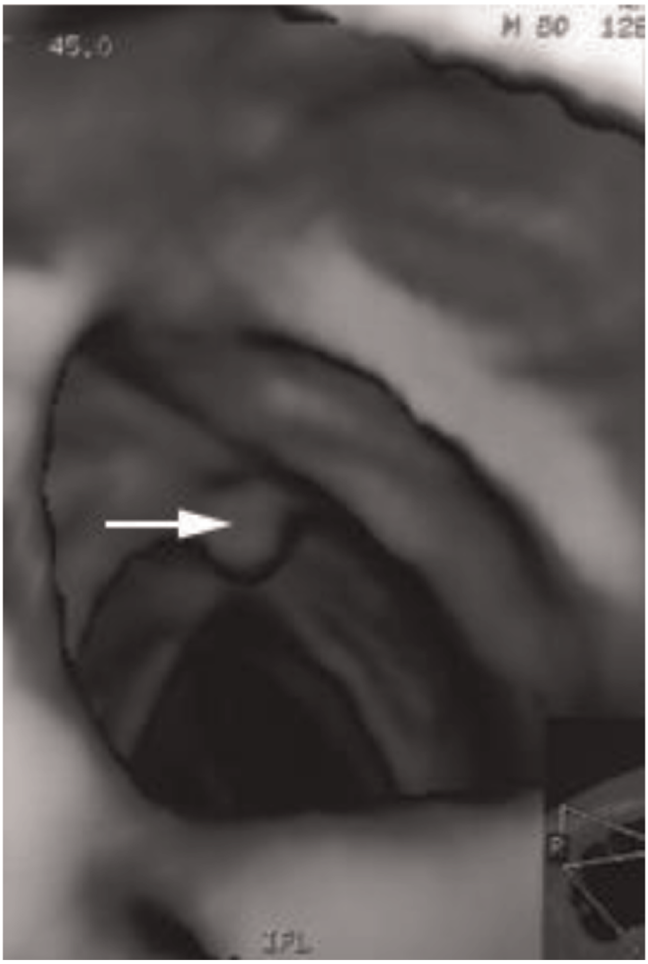

Figure 4.
Colonic polypVirtual colonoscopy shows a small polyp (arrow) in the transverse colon.

- Screening and surveillance for colon cancer
- Lower GI symptoms
- Abnormal imaging
- Evaluation for metachronous or synchronous cancer in patients with colon cancer
- Evaluation of terminal ileum

10.3 Therapeutic

- Foreign body removal
- Removal of polyp

Figure 5.
India ink at polypectomy scar. India ink staining is visible at the site of a polypectomy scar.

- Decompression of sigmoid volvulus or colonic pseudo-obstruction
- Secure hemostasis in lower GI bleed
- Palliative treatment of bleeding or stenosed neoplasms
- Percutaneous endoscopic cecostomy tube placement

10.4 Contraindications

- When the risks of colonoscopy outweigh the expected benefits
- Consent cannot be obtained for non-urgent procedure
- A perforation is known or expected
- Documented acute diverticulitis
- Fulminant colitis

10.5 Important considerations

It is important to consider certain factors in the pre-procedure evaluation of the patient that may affect the successful and safe completion of a colonoscopic examination. These include:

- Anatomic issues, such as history of abdominal and/or pelvic surgeries, recent colonic surgery, presence of a colostomy, and abdominal hernias.
- Comorbidities that may increase sedation-associated risks.
- Comorbidities that may affect to position of the patient properly during colonoscopy or to application of abdominal pressure during the procedure may impact the assistant's ability to do it appropriately.
- The presence of some pacemakers and an implanted cardiac defibrillator need special consideration while using specific management for example. During the application of electrocautery
- The patients on chronic use of certain medications such as narcotics or benzodiazepines, may increase their tolerance of the patient to the effects of sedation
- A history of difficult intubation by anesthesia

10.6 Patient preparation

10.6.1 Diet

Prior to elective colonoscopy, patients need to consume clear liquids or a low-residue diet for at least 1 day. A low-residue diet is low in fiber. So the patients should be instructed to avoid foods that are high in fiber such as whole grains, vegetables,and fruits. Clear liquids include water, tea (without milk), coffee, gelatine, ices, and fruit juices such as grapefruit, apple,and lemonade. Liquids that are red should be avoided as they can be mistaken for blood in the colon and can obscure mucosal details. One trial found that there was no significant difference in preparation quality between patients treated with 4 L of polyethylene glycol who were assigned to receive either a low-residue diet or clear liquids the day prior to the examination [31]. Similarly, a randomized trial with 230 patients who were receiving a sodium sulfate-based preparation assigned patients to either a low-residue diet for breakfast and lunch the day prior to the colonoscopy or to clear liquids [32]. Again, there was no difference between the groups with regard to preparation quality. However, patients in the low-residue arm reported greater satisfaction with the preparation and were less likely to cancel their procedure (9 versus 20 percent). Whether similar results would be seen outside of a randomized trial is not yet known. Our approach is to recommend a low-residue diet for 5 days prior to colonoscopy, with only clear liquids allowed the day prior to the examination.

Patients typically take no food by mouth for four to 8 hours prior to the procedure (sometimes longer if there is known or suspected delayed gastric emptying) and no liquid (other than sips with medications) for 2 hours [33]. Recommendations differ with regard to pre-procedure fasting for elective procedures. The American Society for Anaesthesiology (ASA) guidelines state that prior to a procedure, patients should fast a minimum of 2 hours following clear liquid ingestion or 6 hours for a light meal [34]. By contrast, the American College of Emergency Physicians states "Recent food intake is not a contraindication for administering procedural sedation and analgesia, but should be considered in choosing the timing and target level of sedation" [35]. The usual practice is to follow the ASA approach.

10.6.2 Medications

Most medications may be continued up to the colonoscopy time and can be taken with a small sip of water on the day of the colonoscopy. Some medications may need adjustment prior to colonoscopy, such as medications for diabetes as oral intake is decreased prior to the procedure and there is a risk of hypoglycemia. It is also advised to stop oral iron at least 5 days before the colonoscopy since it can interfere with the results by making the residual feces black, viscous, and difficult to purge.

Decisions regarding the patients taking anti-platelet agents or anti-coagulants, risk- benefit ratio must be carefully assessed keeping in mind the bleeding probability from the procedure while on medications with the probability of thromboembolic event in case of interruption of anti-thrombotic medication [36]. Furthermore, the urgency of the procedure and the availability of alternative tests must be evaluated. So a detailed discussion should be done with the patient and the clinician prescribing the medication for appropriate management plan regarding anti-thrombotic agents.. Aspirin and non-steroidal anti-inflammatory drugs may be continued safely in standard doses in patients having colonoscopy.

Routine antibiotic prophylaxis is not recommended for colonoscopy as the risk of infection is low related to routine diagnostic or therapeutic colonoscopy.

10.6.3 Pre-procedure testing

General recommendation is that routine pre-procedure laboratory testing, electrocardiography, or chest radiography is not required in all patients [37]. Instead, they emphasize testing the selected patients depending on their medical history, physical examination, and risk factors associated with the procedure itself.

The American Society for Gastrointestinal Endoscopy guidelines in 2014 recommended pre-procedure testing in the following settings [37]:

- All the women of childbearing age who give uncertain pregnancy history or whose history is suggestive of a current pregnancy (especially if fluoroscopy is going to be used), should undergo pregnancy testing.

- Coagulation studies for patients who are actively bleeding, or at increased risk of bleeding due to certain medication (e.g. ongoing anti-coagulant use or dual anti-platelet agents), suspected to have bleeding disorder (when there is a history of abnormal bleeding), malnutrition, prolonged biliary obstruction, or other conditions associated with acquired coagulopathies.

- The patients with decompensated heart failure or having respiratory symptoms, chest radiograph should be done.

- The patients with pre-existing clinically significant anemia or who are actively bleeding, or when they are at high risk of significant blood loss during the endoscopic procedure, fresh hemoglobin/hematocrit report should be available.

- Blood typing and cross-match for the patients with active bleeding or anemia who are likely to have a blood transfusion.

- The patients with significant hepatic, endocrine, or renal dysfunction, serum biochemistry is required particularly if need to use medications that may further impair function.

Routinely, most of the centers do not check the coagulation profile of the patients on anticoagulants if the medication has been stopped for an appropriate amount of time before the planned procedure.

10.7 Colonoscopy prepared by sodium phosphate vs. polyethylene glycol

Polyethylene glycol (PEG).(NuLYTELY, Half Lytely, and. GoLYTELY; Braintree Laboratories, Inc., Braintree, MA; Colyte;. Schwarz Pharma, Milwaukee, WI, and MoviPrep; Salix Pharmaceutical, Inc., Morrisville, NC.) and sodium phosphate (NaP) tablets. (Visicol and OsmoPrep Tablets; Salix. Pharmaceuticals, Inc., Morrisville, NC), NaP solution. (Fleet Phospho-soda; C.B. Fleet Company, Inc., Lynchburg, VA), are the most widely. Used agents for colon cleansing. Polyethylene glycol.is an orally administered isotonic solution introduced in 1980 [38]. Since PEG is non-digestible and. Non-absorbable, it cleanses the colon by purging. of intraluminal contents [39]. Because it is iso-osmolar. With plasma, the large volume of PEG does not result in significant fluid shifts. It has been shown to be highly effective when taken as instructed (4 L of PEG.solution). However, the efficacy of standard 4 L PEG outside of clinical trials is compromised by poor patient compliance. The large volume and taste are the main factors that contribute to poor patient compliance and tolerability [40–42], which led to the development of reduced-volume PEG solution. With or without laxatives, sulfate-free, and. Flavored PEG solutions (Half Lytely, NuLYTELY) in an attempt to reduce the sulfate odor and improve the taste [43]. In some studies, split-dose PEG has been more effective than standard 4 L PEG [44, 45].

All patients are asked to avoid eating seeds containing vegetables, berries, fruits, and bread in the week before the colonoscopy. Patients are told to drink 3–4 liters of PEG-EL solution (1.2–1.8 L/h) starting in the afternoon (02:00 pm) on the day before the colonoscopy. Analgesia is given with IV tramadol and phloroglucinol and sedated with midazolam prior to the procedure.

10.8 Assessment of bowel preparationl

Bowel preparations are often described with terms such as unsatisfactory/inadequate, poor, fair, good, and excellent. To standardize descriptions, systems for reporting bowel preparation quality have been developed, including the Boston bowel preparation scale [46]. The score ranges from 0 to 3 (**Figure 6**) for individual colonic segments: the right side of the colon (including the cecum and ascending colon), the transverse colon (including the hepatic and splenic flexures), and the left side of the colon (including the descending colon, sigmoid colon, and rectum). These segment

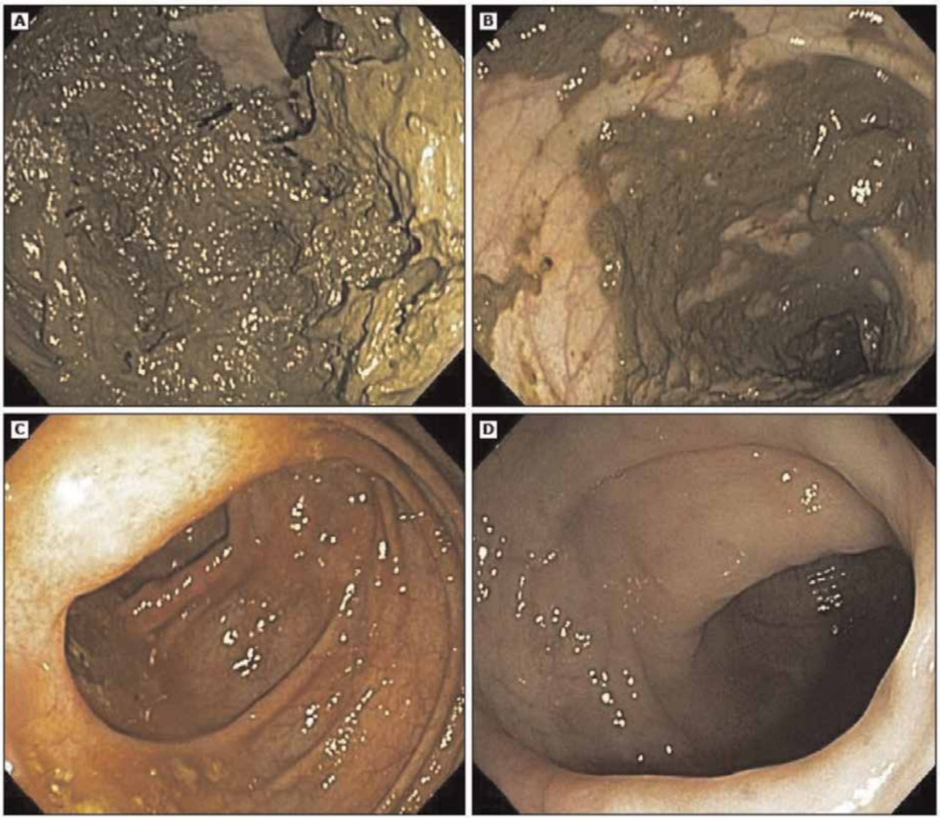

Figure 6.
Boston bowel preparation scale.

scores are summed for a total Boston bowel preparation scale score, which ranges from 0 (poor) to 9 (excellent):

- Score 0: Unprepared colon with mucosa not seen because of solid stool that cannot be cleared
- Score 1: A portion of the mucosa of the colon segment was seen, but other areas of the colon segment were not seen well because of staining, residual stool, and/or opaque liquid
- Score 2: Minor amount of residual staining, small fragments of stool and/or opaque liquid, but most mucosa of the colon segment is seen well
- Score 3: Entire mucosa of colon segment seen well with no residual staining, small fragments of stool, and/or opaque liquid

The BBPS. A: Segment score 0, unprepared colon segment with mucosa not seen because of solid stool that cannot be cleared. B: Segment score 1, portion of mucosa of the colon segment seen, but other areas of the colon segment not well seen because of staining, residual stool, and/or opaque liquid. C: Segment score 2, minor amount of residual staining, small fragments of stool and/or opaque liquid, but mucosa of colon segment seen well. D: Segment score 3, entire mucosa of colon segment seen well with no residual staining, small fragments of stool and/or opaque liquid.

11. Conclusion/awareness to enhance the health care team outcomes

Polyethylene glycol is a polyether compound derived from petroleum having wide range of applications in so many industries including medical, biological, chemical, industrial, commercial and entertainment industries. As a medication, it belongs to laxative class of drugs. It is used in the management and treatment of chronic constipation and fecal impaction due to its water retention property. It is also used for colonoscopy preparation to cleanse the bowel providing clear image for detailed diagnostic evaluation of the colon. It causes side effects which are mostly minor and tolerable. It is an over the counter medication. As such there is increased access to the drug.

Polyethylene glycol is considered to be biologically inert and safe by US FDA. The large percent of population can develop antibodies to PEG because of its ubiquity in multitude of products. Allergic reactions to polyethylene glycol are increasing concern [47, 48].

Healthcare providers need to emphasize the directions for its use and misuse. Patients should be counseled about the onset of action as well as when to discontinue it.

They should also be counseled regarding dietary and lifestyle modifications with treatments.

Nutritionists and nurses can play a role in educating patients on a highly nutritious, fibrous diet and increased fluid intake to help with bowel movement.

This approach will yield improved patient results while minimizing adverse events which are still possible with over-the-counter medication like polyethylene glycol (Level 5).

Author details

Sonia Saleem
Shaikh Zayed Hospital Lahore, Pakistan

*Address all correspondence to: dr.soniasaleem@yahoo.com

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Chapter 4

Blown Film Cooling Numerical and Experimental Investigations

Mohamed Abdel Maksoud

Abstract

The blown film production process involves several processes as extrusion and film cooling. The demand to achieve high productivity with proper film quality has devoted the researchers to investigating in detail, the bubble kinematics, and the thin film cooling process, utilizing the powerful Computational Fluid Dynamics tools, which allows them to deeply study the flow field, and the related heat transfer from the hot thin film to the surrounding mediums, several runs can be done, resulting in huge scientific data, at a minimum cost. In addition to the experimental measurements. The combination of both, reveals better understanding and accurate results.

Keywords: blown film cooling, turbulence modeling, discretization error analysis, experiential measurements, uncertainty analysis, radiation heat transfer

1. Introduction

Blown film production is a widely used polymer processing technology that has been developed considerably during the past decades. The increasing demand to achieve high productivity with high-quality film products has steered more researchers to devote their efforts towards the investigation and development of both equipment and materials used in the blown film industry [1–7].

Blown film production involves several complex processes as the following: -

- **Extrusion**

  During the blown film production process, the polymer pellets are poured inside the hopper and then forced by the screw to flow inside the extruder's barrel. Where the polymer pellets are heated and melted into a viscous fluid, flowing between the extruder's barrel and the rotating screw. The heat conducted from the electric strip heaters through the barrel walls and into the polymer, in addition to the viscous dissipation due to the screw rotation, melts the polymer. The heating process depends on the thermo-physical properties of the polymer under processing, such as High-Density Polyethylene (HDPE), Low-Density Polyethylene (LDPE), etc. The melt exits from a circular orifice at the top of the die forming a bubble. Pressurized air is introduced into the center of the die to inflate the bubble to take the desired dimensions. The bubble radius and thickness variations along the machine direction are controlled by several

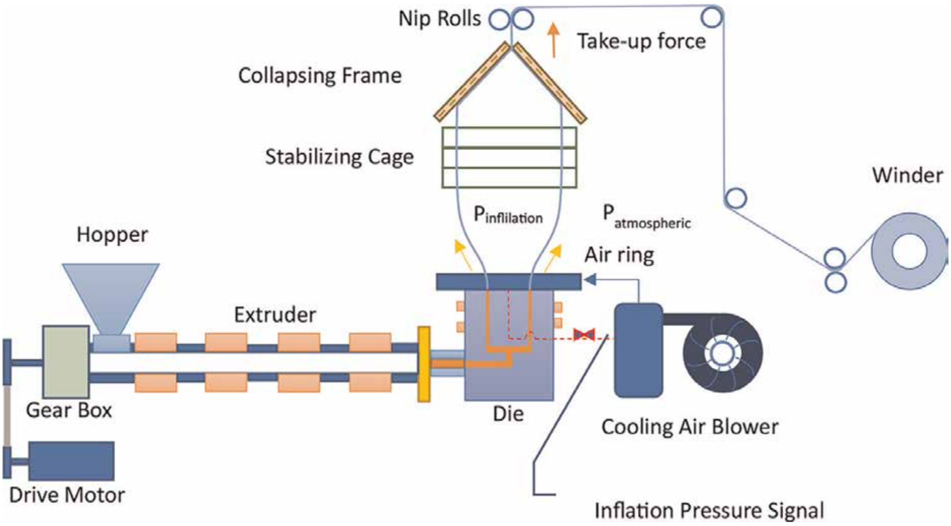

Figure 1.
Typical blown film production system.

operating parameters like the polymer being processed, the size of both the die and the cooling air ring, the internal air pressure, the cooling airflow rate, the melt throughput and the take-off speed, which is defined as the velocity of the blown film as it travels through the nip rollers. **Figure 1** shows a typical blown film production system.

- **Film cooling**

  The cooling process helps the polymer to cool down after it takes the desired shape, dimensions, and final characteristics. Mainly, there are two common air-cooling techniques used in the blown film production process.

  - **The internal bubble cooling (IBC)**

    Where pressurized air is introduced into the center of the die, circulated inside the bubble to cool down the internal bubble surface, and then exhausted outside through the die. This internal cooling air is also used to inflate the bubble.

  - **The external bubble cooling (EBC)**

    The outer surface of the bubble is cooled via an air ring, which is fed from the air blower through several inlet ports distributed on either its side or bottom walls. The air rings are usually designed to be adjustable to control the cooling airflow rate, which exits through the gap between its lips. Air rings would have either single or multiple lips according to the characteristics of the polymer being processed. The air ring has to be aligned correctly with the bubble to guarantee equal distribution of the cooling air around the bubble's body which in turn reduces the possibility to get thickness variation on the bubble lay-flat.

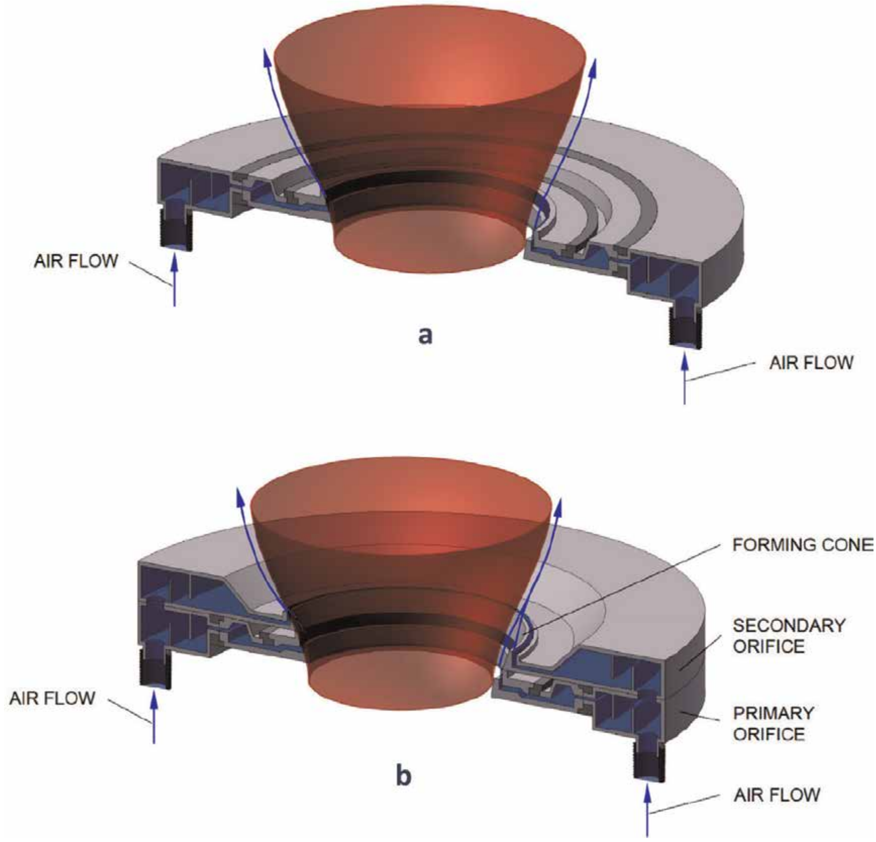

Figure 2.
Typical cross-sections of both single and dual-lip air rings: (a) the single-lip design, (b) the dual-lip design.

One typical air ring design is the single-lip type, which is relatively inexpensive. This design is used with polymer materials that are run with stable bubbles, such as HDPE with a low Melt Index (MI) < 0.1, which is characterized by its long stalk. The MI is defined as the mass of polymer, in grams, flowing in ten minutes through a capillary of a specific length and diameter by applying pressure via prescribed alternative gravimetric weights for alternative prescribed temperatures [8]. While the dual-lip air ring is used with less stable polymer materials that are run with a pocket bubble type. **Figure 2** shows the two air ring designs.

- **Film collapsing**

  The film tube passes through the nip rolls where it is collapsed and flattened. The material of the nip rolls has to have low thermal conductivity to avoid film surface defects. Then the film is wrapped into rolls. The film winder has to be aligned well with the upstream equipment to avoid applying undesirable stresses on the film layers.

- **Production control**

  The blown film production machine shall be equipped with film thickness control and monitoring devices, which measure and correct the thickness variations along the bubble's circumference at the Frost-Line Height (FLH). Besides, bubble stability monitoring and control devices are used to guarantee a stable production process with proper high-quality blown film products.

2. Blown film materials

For blown film products, the desired properties determine the fit for the purpose material needed for a specified application. Blown film extrusion requires good processing characteristics in addition to high-quality products. The most important properties of blown film products are:

- **Optical properties**, such as optical thickness, transmissivity to thermal radiation and haziness.

- **Mechanical properties**, like tensile strength and tear resistance.

- **Thermal properties**, such as solidus and liquidus temperatures, the viscosity of the melt phase, the thermal conductivity, etc.

The ease of the blown film production process can be defined by several material characteristics such as good thermal stability, high melt strength downstream of the die exit, proper head pressures, and film surface clarity. Finally, cost optimization is a key factor.

Polyethylene (PE) is considered the best polymer for most blown film production applications. It has good water resistance, is lightweight, has an acceptable balance of flexibility and strength, and can have good clarity. Moreover, PE is considered easy to heat-seal and extrude, at a low cost. In addition to the above-mentioned common properties of PE, it is a polymer that is well understood scientifically, this led to well-designed and controlled polymerization techniques to yield specific property values over a very wide range. Particularly, PE grades can be produced with better clarity, acceptable higher strength than average, more flexibility, etc. Within the broad family of polyethylene, different types are used in the blown film extrusion industry as follows:

2.1 Polyethylene (PE)

Based on a chemical point of view, it is considered the simplest polymer. PE is polymerized from an ethylene monomer, which contains a carbon chain backbone with two hydrogen atoms bonded to each carbon atom. Individual chains or molecules would contain tens to hundreds of carbon atoms. Chains can be branched or linear, based on how the polymer was synthesized. There are several synthesizing techniques for polyethylene production [9]. All PE grades are characterized by high specific heat values, which means a relatively slow heat removal from the polymer during processing. The specific heat of PE is about 2 kJ/kg·K compared to approximately 1 kJ/kg·K for most other different polymers. Consequently, cooling towers for PE-blown film production are extremely tall. It requires enough time to release the remaining heat from the two layers of the film moving through the nip rollers to prevent them from blocking.

2.2 Low-density polyethylene (LDPE)

Polyethylene is often classified according to its density. When a polymer cools from the melt state and solidifies, some of the chains would organize into denser, highly ordered crystalline zones. LDPE has a density that varies between 0.93 and 0.91 g/cm^3. It has good processing characteristics and does not require too much motor torque to rotate the screw. Besides, it melts at approximately 105–115°C. On the other hand, the existence of a wide range of branching yields a bubble with high melt strength and an adequately wide processing window. The LDPE films possess a good combination between elongation and strength in addition to good flexibility.

2.3 High-density polyethylene (HDPE)

Is synthesized by a different method that is used for LDPE. It is produced with linear chains. HDPE is polymerized with a few amounts of comonomer and results in a few short-chain branches. Which are placed intentionally along the main chain to get a polymer with good processability. A high degree of linearity produces a polymer with a high percentage of crystallinity and consequently high density. HDPE has a density range of 0.96–0.93 g/cm^3. HDPE melts at relatively higher temperatures 130–135°C. Furthermore, it has a narrower processing window. In addition, it consumes a high screw torque.

One of the clearest differences between processing HDPE and LDPE is that, HDPE with a low $MI < 0.1$ runs usually with a high FLH. Which is about 8–10 times the bubble's initial diameter at the die. Bubble instabilities would exist during the processing of HDPE due to its lower melt strength. Delaying bubble transverse stretching until the melt gets cooler i.e., at higher FLH, the bubble becomes more stable. In addition, HDPE-blown films are characterized by their high stiffness and strength. Hence, there is persistent progress to produce HDPE film products with small thicknesses.

2.4 Linear low-density polyethylene (LLDPE)

LLDPE is considered a variation of HDPE. It is manufactured in the same way with many comonomers, such as octane or hexene. The existence of a comonomer in the chain results in shorter-chain branches of a certain length. The density of LLDPE varies between 0.93 and 0.88 g/cm^3 and has a melt temperature of 115–125°C. Processing of LLDPE needs high screw torque with a grooved feed throat similar to HDPE. However, outside of the die, it is processed with a pocket bubble profile similar to LDPE, even though the melt strength is lower than that of LDPE. To guarantee proper bubble stability, dual or multiple-lip air rings are used for high cooling airflow rates. The LLDPE film's final properties are considered a combination of those of LDPE and HDPE.

3. Machine setup requirements

The setup of the blown film production machine requires the adjustment of the following operating parameters, which shall be performed by skilled technicians.

- The temperature of the heaters according to the specified melting temperature of the polymer being processed like HDPE, LDPE, LLDPE, etc.

- The inflation pressure shall be higher than the atmospheric pressure.
- The inflation air volume has to be adjusted with the inflation pressure to get the desired bubble's profile and characteristics:
    - The Blow-Up ratio (BUR), is the ratio of the bubble's diameter at the FLH to that at the die exit.
    - The FLH is the height at which, the bubble's radius and the film thickness become fixed. Moreover, it corresponds to the optimum cooling and desired film properties.
- The Take-Up Ratio (TUR) is defined as the ratio between the film velocities at the FLH to that at the die exit.

The final film dimensions and characteristics are obtained by performing fine-tuning between the above-mentioned parameters.

4. Numerical and experimental investigations

During the blown film production process, the molten polymer exits the die through a circular orifice at its top, forming a bubble with varying radius and thickness. The bubble body is subjected to the differential pressure between the internal inflation pressure, and the external atmospheric pressure, the take-up force which pulls the film up in the machine direction, see **Figure 1**, and the cooling air which cools down the hot bubble and transforms it to the solid phase after taking the desired dimensions and characteristics. The complex processes associated with the production of the blown film reveal different related research fields, which can be categorized as follows:

- **Rheology**, where the bubble kinematics are mainly considered [1, 10, 11]. The bubble's profile and the film thickness variation along the machine direction are investigated numerically and experimentally for different polymer viscosity modeling and different operating conditions.
- **Aerodynamics of the blown film cooling**, where the flow field around the bubble's surfaces and the corresponding heat transfer are investigated. In addition to, the air ring design and its influence on the generated flow field and the cooling efficiency [5, 7, 12–14].
- **Experimental measurements** are applied to investigate the bubble's profile, the surface temperature, and the film thickness variations with the machine direction, the film's mechanical and optical properties, and the bubble's stability monitoring and control [2, 15].
- Recently, due to the progress that occurred in the field of blown film production, the dependency of the bubble kinematics on the film surface temperature distribution and the temperature gradients inside the film thickness, some researchers devoted their efforts to combining the blown film aerodynamics with the bubble kinematics [6, 16–19]. Besides, the bubble's stability during the production process is also investigated as an important monitoring issue [20].

5. Numerical modeling guidelines

The use of Computational Fluid Dynamics (CFD) as a simulation tool enables the researchers to study in detail the physical phenomena associated with the flow pattern and the related heat transfer process. Besides, several runs and design optimizations can be done in a short time and at a minimum cost. The calculation domain in blown film cooling can be divided into three distinct zones namely, the external cooling air domain, the bubble body domain, and the internal air\cooling air domain as illustrated in **Figure 3**.

5.1 The turbulent cooling airflow numerical modeling

Blown film cooling airflow is mostly turbulent. The selection of the more accurate turbulence model, the required near-wall treatments, in addition to the mesh size requirements while constructing the calculation domain matters. The turbulence modeling passed several modifications during the past decades, the basis of the first step was the time averaging of the governing equations.

5.1.1 The Reynolds Averaged Navier Stokes (RANS) Equations

Starting with the incompressible, constant-property flow. The conservation equations of mass and momentum take the following forms:

$$\frac{\partial u_i}{\partial x_i} = 0 \tag{1}$$

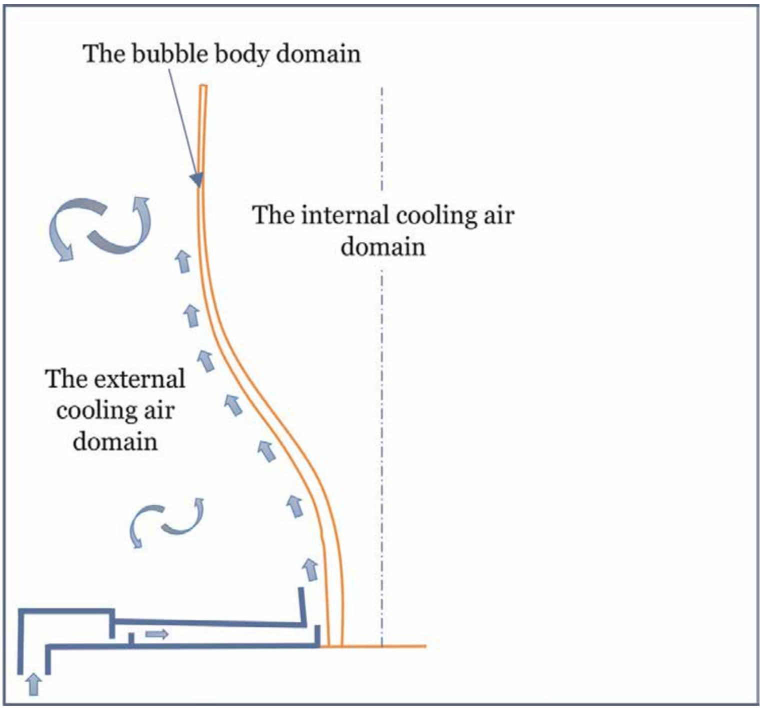

Figure 3.
Blown film cooling calculation domains.

$$\rho\frac{\partial u_i}{\partial t}+\rho u_j\frac{\partial u_i}{\partial x_j}=-\frac{\partial p}{\partial x_i}+\frac{\partial t_{ji}}{\partial x_j} \tag{2}$$

The vectors x_i and u_i, are position and velocity, t is time, p is static pressure, ρ is density and t_{ij} is the viscous stress tensor defined by

$$t_{ij}=2\mu s_{ij} \tag{3}$$

Where the strain-rate tensor is defined by:

$$s_{ij}=\frac{1}{2}\left(\frac{\partial u_i}{\partial x_j}+\frac{\partial u_j}{\partial x_i}\right) \tag{4}$$

Applying time averaging on Eqs. (1) and (2), yields the well-known Reynolds Average Equations of motion in its conservation form, viz.

$$\frac{\partial U_i}{\partial x_i}=0 \tag{5}$$

$$\rho\frac{\partial U_i}{\partial t}+\rho U_j\frac{\partial U_i}{\partial x_j}=\frac{-\partial P}{\partial x_i}+\frac{\partial}{\partial x_j}\left(2\mu S_{ji}-\ \rho\overline{u_i'u_j'}\right) \tag{6}$$

The only difference between the instantaneous and the time-average momentum equations is the existence of the correlation $\overline{u_i'u_j'}$, which is the time-average rate of momentum transfer because of turbulence. Moreover, the quantity $-\rho\overline{u_i'u_j'}$ is the Reynolds-stress tensor. The computation of this term is the fundamental problem of turbulence modeling. The RANS models can be closed as follows:

5.1.2 The Reynolds-Stress Model (RSM)

In this model, the specific Reynolds stress tensor is directly solved via transport equations. However, modeling is still needed for several terms in the transport equations. The RSM is more advantageous in complex three-dimensional turbulent flows possessing swirl and large streamline curvature. The RSM model is more complicated to converge than eddy viscosity models. Besides, it is computationally intensive.

5.1.3 The Eddy Viscosity Models, (Boussinesq hypothesis)

The Reynolds stresses are modeled by introducing turbulent or eddy viscosity, ν_T.

$$\tau_{ij}=\nu_T\left(\frac{\partial\bar{u}_i}{\partial x_j}+\frac{\partial\bar{u}_j}{\partial x_i}\right)-\frac{2}{3}k\delta_{ij} \tag{7}$$

Where the Kronecker delta function takes the form:

$$\delta_{ij}=\begin{cases}1, & i=j\\ 0, & i\neq j\end{cases} \tag{8}$$

The Boussinesq hypothesis is suitable for simple turbulent shear flows such as boundary layers, round jets, channel flows and mixing layers, etc.

Noting that all turbulence models have empiricism because.

- Equations cannot be closed based on fundamental principles.
- Calibration is required to set up the model constants.

Based on the Boussinesq hypothesis, the dimensions of the turbulent kinematic viscosity ν_T is (length^2/time) i.e., according to dimensional analysis, it can be considered as the product of velocity scale by a length scale.

Mainly there are four categories of eddy viscosity models:

- Algebraic or zero-equation models.
- One-equation models.
- Two-equation models.
- Stress-transport models.

We will move forward to the two-equation models.

5.1.3.1 Two-equation models

The improvement in computer capabilities since the 1960s allowed looking forward to utilizing more complicated models. For example, the turbulence models based on the equation formulating the turbulence kinetic energy have become the basis of modern research in turbulence modeling. The two-equation models are eddy-viscosity-based. That they retain the Boussinesq eddy-viscosity approximation.

Prandtl (1945) defined the turbulence kinetic energy (per unit mass) of turbulent fluctuations.

The specific turbulence kinetic energy, k is given by:

$$k = -\frac{1}{2}\tau_{ii} = \frac{1}{2}\overline{u_i' u_i'} = \frac{1}{2}\left(\overline{u'^2} + \overline{v'^2} + \overline{w'^2}\right) \tag{9}$$

Based on the dimensional arguments, the kinematic eddy viscosity can be written as a product of a velocity scale by a length scale.

$$\nu_T = C k^{1/2} \ell \tag{10}$$

Where C is a constant.

The transport equation for the turbulence kinetic energy k takes the following form:

$$\frac{\partial k}{\partial t} + U_j \frac{\partial k}{\partial x_j} = \tau_{ij}\frac{\partial U_i}{\partial x_j} - \varepsilon + \frac{\partial}{\partial x_j}\left[\nu \frac{\partial k}{\partial x_j} - \frac{1}{2}\overline{u_i' u_i' u_j'} - \frac{1}{\rho}\,\overline{p' u_j'}\right] \tag{11}$$

The quantity ε is the dissipation rate per unit mass and is defined by:

$$\varepsilon = \nu \overline{\frac{\partial u_i'}{\partial x_k}\frac{\partial u_i'}{\partial x_k}} \tag{12}$$

At this point, there are two unknowns, which are the turbulent length scale, ℓ and the dissipation rate, ε. Both properties are assumed solely functions of turbulence only. The dimensional arguments of [Taylor (1935)] showed that:

$$\varepsilon \sim k^{3/2}/\ell \tag{13}$$

The system of equations still needs a prescription for the turbulence length scale to be closed. Hence, the following version of the turbulence kinetic energy equation is applied in all turbulence energy equation-based models and takes the following simplified form:

$$\frac{\partial k}{\partial t} + U_j \frac{\partial k}{\partial x_j} = -\tau_{ij} \frac{\partial U_i}{\partial x_j} - \varepsilon + \frac{\partial}{\partial x_j}\left[(\nu + \nu_T/\sigma_k)\frac{\partial k}{\partial x_j}\right] \tag{14}$$

Where σ_k is a closure coefficient.

Moreover, Prandtl (1945) introduced a closure coefficient to Eq. (13) called C_D

$$\varepsilon = C_D k^{3/2}/\ell \tag{15}$$

In any two-equation model, a second transport equation is needed to close the system.

The different two-equation-based models can be summarized as follows:

5.1.3.2 The $k - \omega$ model

The specific dissipation rate ω, with dimensions of (1/time), is selected as the second variable to close the system of equations. The developers of the $k - \omega$ model after Kolmogorov added a production term like Wilcox [21], Kok [22], and Hellsten [23]. The updated version of the $k - \omega$ model improved predictions of Wilcox model [21], for strongly separated and free shear flows. The $k - \omega$ showed good accuracy in predicting free shear, wall-bounded and complicated separated flows when compared with experimental data.

5.1.3.3 The $k - \varepsilon$ model

The $k - \varepsilon$ model is one of the famous two-equation models. It was developed by several authors such as Launder and Sharma [24]. The formulation of the exact second transport equation started by taking the moment of the Navier-Stokes equation, and resulted in the well-known standard $k - \varepsilon$ model [25]. Yakhot et al. [26] used techniques from the Renormalization Group (RNG) theory and developed the standard $k - \varepsilon$ model to be the well-known RNG $k - \varepsilon$ model. The new model uses a differential equation to calculate the turbulent viscosity, an analytical formula to calculate the turbulent Prandtl number and a modified new coefficient to improve the accuracy for rapidly strained flows.

5.1.3.4 The baseline (BSL) $k - \omega$ model

This model is formulated to combine the advantages of the $k - \varepsilon$ model, which performs well for the outer wake region, and the $k - \omega$ model, which is more accurate for the inner zone of the boundary layer [27]. A blending function F_1 is used and formulated to gradually change from one in the near-wall region to zero away from the walls. The BSL model is formulated by transforming the $k-\varepsilon$ model into a $k-\omega$ model. Then, assuming ς_1 represents any constant of the original $k-\omega$ model, ς_2 any constant of the transformed $k-\varepsilon$ model and ς is any constant of the new BSL formulation. The baseline model retains the advantages of the $k-\omega$ model for wall-bounded flows without freestream dependency.

5.1.3.5 The shear stress transport (SST) $k-\omega$ model

The formulation of the SST $k-\omega$ model is the same as the BSL $k-\omega$, with a modified definition of the turbulent viscosity ν_T to control the overproduction of turbulent shear stress in adverse pressure gradient flows, a blending function F_2 is used [27]. The SST $k-\omega$ model has very good behaviour in separation flows and adverse pressure gradients. Nevertheless, it would produce a bit too large turbulence levels in regions with large normal strains, such as stagnation regions.

5.2 The energy equation

In addition to the previously described turbulence models, which are formulated to obtain the average velocity and pressure fields, the energy equation is solved to obtain the corresponding average temperature field.

The general form of the turbulent transport of the total energy E takes the following form:

$$\frac{\partial(\rho E)}{\partial t}+\frac{\partial(\rho U_i E)}{\partial x_i}=\frac{\partial}{\partial x_i}\left[\alpha\mu_{eff}C_p\frac{\partial T}{\partial x_i}+U_i\left(\tau_{ij}\right)_{eff}\right]-\frac{\partial(PU_i)}{\partial x_i} \tag{16}$$

Where α is the inverse Prandtl number.

The polymer flow is simulated as a fluid moving through a virtual converging channel in the machine direction, where it is transformed from melt to solid phase due to the cooling process.

The energy equation for the polymer film takes the following form:

$$\frac{\partial\rho H}{\partial t}+\nabla.\left(\rho\vec{u}H\right)=\nabla.(K.\nabla T)+S \tag{17}$$

Where H is the material enthalpy $H=h+\Delta H$, $\vec{u}$ is the velocity vector, and S is a source term.

The thermal boundary condition at the film surface:

$$-K\frac{\partial T}{\partial n}\bigg|_{Fs}=(HTC_C+HTC_R)(T_{Fs}-T_\infty) \tag{18}$$

Further, near-wall treatments and turbulence kinetic energy production limiters are applied to improve the accuracy of the two-equation models [25, 28].

5.3 Blown film thermal radiation characteristics

Blown films' thickness varies between a few millimeters at the die, to a few microns at the FLH, **Figure 4** shows that thin polyethylene films are highly

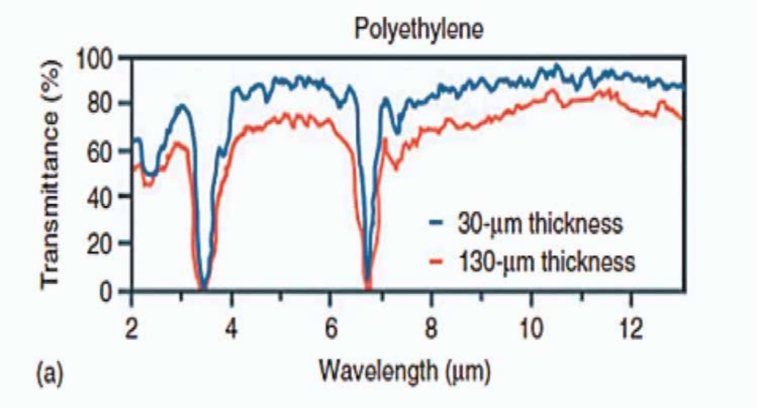

Figure 4.
Transmissivity versus wavelength for polyethylene film at 30 and 130 µm thicknesses [29].

transparent to infrared thermal radiation except at 3.4 & 6.7 µm wavelengths, where their transmissivity is nearly dropped to zero. As illustrated, a 30 µm polyethylene film has a maximum of 90% transmissivity, and a 130 µm film is 70% [29]. According to Beer-Lambert's law [30, 31], transmissivity takes the following form:

$$\zeta(x) = (1 - \varrho)^2 e^{-\gamma n(x)} \tag{19}$$

Polyethylene films are characterized by low reflectivity ϱ about 0.05 [32]. Consequently, the transmissivity $\zeta(x)$ would approximately correlate with the film thickness as follows:

$$\zeta(x) = 0.9 e^{-\gamma n(x)} \tag{20}$$

Where n: is the film thickness and γ is the absorption coefficient, a typical value of 0.045 (Mils^{-1}) is found to be a reasonable correlation value. The term γn is defined as the optical thickness. Noting that (one Mil) equals (0.001 in).

The accurate numerical simulation requires a proper treatment of the radiation heat transfer from, into, and through the semitransparent film. The Discrete Ordinates (DO) radiation model [28], is used to include radiation heat transfer into consideration.

5.4 Discretization error analysis

CFD simulations with the implementation of advanced numerical modeling tools allow a better physical-based representation of actual complex flows. The uncertainty of the numerical simulations' accuracy remains very important to ensure its strength. Even though, there is good agreement with the experimental results. The implementation of the Richardson extrapolation method [33, 34], and the Grid Convergence Index (*GCI*) methodology has been evaluated over many CFD cases. It permits a proper quantification of the uncertainty present in grid convergence [35–37].

6. Experimental measurements

The blown film production process involves several necessary measurements, the cooling air flow rate, the melt throughput, the ambient conditions, the air ring and the head walls temperatures, the bubble's profile, and the film thickness and temperature variations along the machine direction. Moreover, some mechanical and optical properties are measured as, tensile strength, tear resistance, and haziness. Besides, the bubble's stability is also monitored during the production process.

6.1 The inlet cooling airflow rate calculations

The air blower provides air into the air ring through uniformly distributed inlet ports. Two methods can be used to calculate the inlet cooling air flowrate:

- By measuring the velocity profile along the pipe's radius and calculating the flow rate by integration.

- By using a measuring tube to have a fully developed flow, and measuring the maximum velocity at the centerline as per the following algorithm:

Recalling the Direct Numerical Simulation (DNS) results [38], the turbulent pipe flow exhibits a universal behavior. Where the mean streamwise velocity varies logarithmically with the normal distance to the wall.

The law of the wall takes the following form for turbulent pipe flows:

$$\frac{u(r)}{u_\tau} = \frac{1}{\kappa} ln \frac{u_\tau(R-r)}{\nu} + B \tag{21}$$

Where $\kappa \approx 0.41$, and $B \approx 5$ for smooth surfaces.

R: is the pipe radius, r: is the normal distance from the pipe centerline, and u_τ: is the friction velocity, $u_\tau = \sqrt{\frac{\tau_w}{\rho}}$.

The pipe average velocity is calculated as follows [38]:

$$\bar{u} = \frac{Q}{A} = \frac{1}{\pi R^2} \int_0^R u_\tau \left(\frac{1}{\kappa} ln \frac{u_\tau(R-r)}{\nu} + B \right) 2\pi r dr \tag{22}$$

$$\frac{\bar{u}}{u_\tau} \approx 2.44 \, ln \frac{u_\tau R}{\nu} + 1.34 \tag{23}$$

The ratio between the "pipe's average to maximum" air velocity can be formulated as:

$$\frac{\bar{u}}{u_0} \approx \left(1 + 1.3\sqrt{f}\right)^{-1} \tag{24}$$

Where f is the friction factor, and u_0 is the centerline maximum velocity.

$$f = 0.316 \, Re_D{}^{-0.25}, \qquad 4000 < Re_D < 10^5 \tag{25}$$

Where the Reynolds number is based on the pipe's hydraulic diameter, D and the average velocity, $\overline{u}$ is defined as:

$$Re_D = \frac{\overline{u} D}{\nu} \tag{26}$$

On the other hand, one of the most widely used temperature-measuring non-contact devices is the Infrared (IR) pyrometer [39, 40]. By identifying the spectral bands, radiation thermometers can be selected and used to measure both the surface and below-surface temperatures of plastic films which are characterized by their transparency to infrared radiation [29]. For an opaque surface with unknown emissivity, the device can be calibrated via thermocouples such as the K-type [41].

Regarding the bubble's profile and film thickness variations with the machine direction, Liu et al. [42] performed experimental investigations on blown films of LDPE, LLDPE and HDPE materials. During the experiments, a video camera was used to measure the bubble's shape and the film velocity, while a remote infrared sensing device measured the film surface temperature.

Combinations between experimental and numerical investigations have been studied by several authors as the following:

Zatloukal and Vlček [6] developed a model to describe bubble formation by applying variational principles. The basis of this model is that the bubble's shape shall satisfy the minimum energy requirements. The proposed simple analytically resolvable equation with four physical parameters had been derived from the variational principles, successfully describing different bubble profiles, including the HDPE wineglass shape, taking the take-up force and the internal bubble pressure into consideration. The results were in good agreement with the experimental data. Also, Zatloukal and Kolarik [19] performed experiments on a 9-layer blown film to investigate how the cooling process affects the film surface temperature variation, the bubble's shape and the product's mechanical properties. The obtained experimental data followed by the theoretical analysis were used to evaluate three different convective heat transfer models. Both internal and external bubble cooling techniques were applied. It was found that only the Muslet & Kamal heat transfer model was correctly able to capture the physical characteristics of the heat transfer between the bubble and the cooling air, especially in the case of a very high FLH. Moreover, Ismail, ME [43] performed numerical and experimental investigations on the external cooling of an HDPE bubble with a new design of a counter flow air ring design. The performance of four different two-equation-based turbulence models is compared to the experimental results of the measured air velocity via a five-hole pitot tube.

6.2 Measurements uncertainty

No measurement done as a part of any scientific research, no matter how carefully we look, can be considered exact. The quantification of the uncertainty associated with the measurements is very important to identify the value of the measurand precisely [44].

7. Conclusions

Blown film cooling is a very important, and complicated process. The enhancement of the cooling efficiency would improve productivity and film quality via the following:

- Applying new cooling techniques.

- Proper understanding of the thermal characteristics of the film, turbulence models, and utilization of the powerful CDF tools. The accurate selection of the most advanced turbulence models, with the supporting discretization error analysis, matters.

- Accurate measurements, supported by the uncertainty analysis, support the scientific investigations and verify the numerical analysis.

Nomenclatures

A	Cross-sectional area, [m^2]
C, C_D	Constants, [—]
C_P	Specific heat at constant pressure, [J/kg.K]
D	Diameter, [m]
E	Total Energy, [J]
f	Darcy friction coefficient, [—]
F_1	Blending function used in the BSL & SST $k-\omega$ models formulation, [—]
F_2	Blending function used in the SST $k-\omega$ model formulation, [—]
h	Sensible enthalpy, [J]
H	Material enthalpy, [J]
HTC	Heat transfer coefficient, [W/m^2.K]
i,j	Unit vectors in x and y directions, [—]
$\boldsymbol{k}$	Turbulence kinetic energy, [m^2/s^2]
K	Thermal conductivity, [W/m.K]
$\boldsymbol{\ell}$	Turbulence length scale, [m]
n	Unit vector normal to the film surface direction, [—]
$n(x)$	Film thickness in the machine direction, [m]
p	Instantaneous pressure, [Pa]
$\boldsymbol{p}'$	Fluctuating static pressure, [Pa]
P	Average static pressure, [Pa]
Q	Volumetric flow rate, [m^3/s]
r	Radial distance, [m]
R	Radius, [m]
Re	Reynolds number, [—]
s_{ij}	Instantaneous strain-rate tensor, [1/s]
S	Shear rate, [1/s]
S_{ij}	Mean strain-rate tensor, [1/s]
t	Time, [s]
T	Temperature, [K]
t_{ij}	Instantaneous viscous stress tensor, [Pa]
u,v	Instantaneous velocity component in the x and y directions, [m/s]
$\boldsymbol{u_i}$	Instantaneous velocity in tensor notation, [m/s]
u'	Fluctuating velocity component in x-direction, [m/s]
$\boldsymbol{u_i'}$	Fluctuating velocity in tensor notation, [m/s]
$\bar{\boldsymbol{u}}$	Fully developed crossectional average air velocity inside the inlet flowrate measuring tube, [m/s]
$\boldsymbol{u_\tau}$	Friction velocity, [m/s]
$\boldsymbol{u_0}$	Fully developed centerline air velocity inside the inlet flowrate measuring tube, [m/s]

$u(r)$	Radial air velocity inside the inlet flowrate measuring tube, [m/s]
U	Mean velocity component in the x-direction, [m/s]
$\boldsymbol{U_i}$	Mean velocity in tensor notation, [m/s]
v'	Fluctuating velocity component in the y-direction, [m/s]
w'	Fluctuating velocity component in the z-direction, [m/s]
$\boldsymbol{x_i}$	Position vector in vector notation, [—]
α	Inverse Prandtl number, [—]
$\boldsymbol{\gamma}$	The blown film absorption coefficient, [m^{-1}]
$\boldsymbol{\delta_{ij}}$	Kronecker delta function, [—]
$\boldsymbol{\varepsilon}$	Dissipation rate, [$\mathrm{m}^2/\mathrm{s}^3$]
ς	Represents any constant of the new BSL $k-\omega$ model, [—]
ς_1	Represents any constant of the original $k-\omega$ model, [—]
ς_2	Represents any constant of the transformed $k-\varepsilon$ model, [—]
$\zeta(\boldsymbol{x})$	Transmissivity of the blown film, [—]
$\boldsymbol{\kappa}$	Von Karaman constant, [—]
$\boldsymbol{\nu}$	Kinematic viscosity, [m^2/s]
$\boldsymbol{\nu_T}$	Turbulence kinematic viscosity, [m^2/s]
$\boldsymbol{\rho}$	Density, [$\mathrm{kg/m}^3$]
$\boldsymbol{\varrho}$	The reflectivity of the blown film surface, [—]
μ	Molecular viscosity, [Pa.s]
$\boldsymbol{\sigma_k}$	Closure coefficient, [—]
$\boldsymbol{\tau_{ij}}$	Specific Reynolds stress tensor, [$\mathrm{m}^2/\mathrm{s}^2$]
$\boldsymbol{\tau_w}$	Surface shear stress, [Pa]
$\boldsymbol{\omega}$	Specific dissipation rate, [1/s]
C	Convective
eff	Effective
R	Radiative
Fs	Film surface
∞	Ambient conditions
$\bar{\ }$	Average value
BUR	Blow-Up Ratio
CFD	Computational Fluid Dynamics
DNS	Direct Numerical Simulation
EBC	External Bubble Cooling
FLH	Frost-Line Height
GCI	Grid Convergence Index
HDPE	High-Density Polyethylene
IBC	Internal Bubble Cooling
IR	Infrared
LDPE	Low-Density Polyethylene
LLDPE	Linear Low-Density Polyethylene
MI	Melting Index
PE	Polyethylene
RANS	Reynolds Average Navier-Stokes
RNG	Renormalization Group
RSM	Reynolds Stress Model
SST	Shear Stress Transport
TUR	Take-Up Ratio

Author details

Mohamed Abdel Maksoud[1,2]

1 Mechanical Power Engineering, Mansoura University, Egypt

2 Egyptian Petroleum Sector, Egypt

*Address all correspondence to: m_abdulmaksoud@hotmail.com

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Chapter 5

Synthesis of Polyethylene-Based Materials, Ion Exchanger, Superabsorbent, Radiation Shielding, and Laser Ablation Applications

Ahmet Hakan Yilmaz, Bülend Ortaç, Saliha Mutlu and Sevil Savaskan Yilmaz

Abstract

Polyethylene (PE), ion exchangers and membranes, super absorbents, radiation shielding, and laser ablation are covered initially. In the second part, studies on polyethylene-based ion exchangers and membranes are given. Ion exchangers and membranes chemically modify copolymers to exchange ions in the electrolyte solution. The third segment covers research on polyethylene-based super absorbent copolymers and composites for water retention and heavy metal contamination removal. Super absorbent polymers are hydrophilic, water-insoluble polymers that absorb plenty of water. With their expansion capacity and user-friendliness, super absorbents are widely used in various fields such as biomedicine, drug distribution, personal care products, batteries, tissue engineering, construction, food packaging, heavy metal separation, electronics, cables, cosmetics, and agriculture. Polyethylene-based polymeric composites are used more to minimize radiation. PE's high hydrogen concentration absorbs neutrons, making it a radiation shield. Block copolymers of polystyrene-b-poly-ethyleneglycol and boron nitride particles can make selenium dioxide a structural material and radiation barrier when paired with nanostructures. The fourth section examines the optical and conductivity properties of laser-ablated polyethylene nanocomposites with metal oxide nanoparticles. Laser ablation polishes metal, transparent materials, composites with surface and interior changes, and nanomaterials. Polymer laser ablation improves surface modification and thin layer deposition. Laser wavelength affects UHMWPE wettability.

Keywords: polyethylene, ion exchanger, superabsorbent, radiation shielding, laser ablation applications, polyethylene-based materials

1. Introduction

Polyethylene (PE), a polymer of great significance and utility, has been subject to extensive research as a plastic material [1–6]. PE is widely recognized as a highly advantageous plastic material for commercial applications due to its exceptional mechanical properties, notable flexibility, commendable chemical resistance, lightweight nature, favorable thermal stability, and cost-effectiveness [7, 8]. The size of the global PE market was assessed at USD 105.77 billion in 2022 and is anticipated to increase from USD 110.23 billion in 2023 to USD 146.20 billion by 2030, demonstrating a compound annual growth rate (CAGR) of 4.1% throughout the forecast period [9]. PE is utilized in various industries, such as agriculture [10], manufacturing [11, 12], medicine [13, 14], construction [15], packaging [16], energy [17], outdoor items [18], and others, owing to its advantageous technical properties and cost-effectiveness in comparison with alternative materials.

PE can be classified into two primary categories: High-density polyethylene (HDPE) is characterized by its elevated density and notable resistance to elevated temperatures. This material is commonly employed due to its notable mechanical strength and chemical resistance. Polyvinyl chloride (PVC) is a resilient and environmentally sustainable polymer that finds extensive utilization in various domains, including water pipe systems, water storage containers, plastic bottles, toys, and packaging materials. Low-density polyethylene (LDPE) is a variant of PE characterized by its reduced density and enhanced flexibility in its molecular structure. Seamless plastic bags are commonly employed in a range of applications, including plastic films, closures, coatings, and diverse packaging materials. PE offers several advantages: The polymer in question is characterized by its widespread availability and relatively affordable cost. Due to its lightweight nature and malleability, this material finds extensive utilization across a wide range of applications. The material exhibits resistance to various chemicals and demonstrates waterproof characteristics. Recycling is a straightforward process that allows for the reuse of materials. However, PE also possesses certain limitations: The material exhibits sensitivity to ultraviolet (UV) radiation and elevated temperatures, thereby leading to the occurrence of cracks and alterations in coloration when employed in outdoor environments. At low temperatures, it has the potential to exhibit brittleness. The process of biodegradation is characterized by a lengthy duration, resulting in the potential for environmental pollution. Hence, the significance of recycling and the utilization of PE in environmentally sustainable practices cannot be overstated. The mitigation of PE's environmental impact can be achieved through the implementation of measures, such as the reduction of single-use plastic products and the enhancement of recycling infrastructure.

Ion-exchange polymers refer to a kind of artificial ion-exchange resin that is composed of cross-linked organic copolymers. These copolymers possess functional groups that have been incorporated into a copolymer matrix through a chemical modification process. These functional groups have the ability to dissociate and exchange ions with ions derived from the surrounding electrolyte solution. Spherical granules, which are insoluble in both water and organic solvents, are often acquired [19]. Ion-exchange membranes (IEMs) play a significant role in a wide range of technologies, primarily due to their crucial characteristics of perm-selectivity and resistance. The membranes can be categorized into two types: heterogeneous membranes, which are created by combining an ion-exchange resin and a thermoplastic polymer, and homogeneous membranes, which possess ion-exchange groups that are chemically attached to a cross-linked backbone [20, 21]. Membranes are classified as anion-exchange

(AEM) and cation-exchange (CEM) membranes based on their level of selectivity. The utilization of ion-exchange (IE) procedures has demonstrated its effectiveness in the removal of natural organic matter (NOM), heavy metal ions, and lanthanides. An examination was conducted to examine the sorption and separation of lanthanides using ion exchangers of different types [22, 23]. A set of anion-exchange membranes (AEMs) was made utilizing a straightforward casting approach, employing polyvinyl alcohol/branched polyethyleneimine (PVA/BPEI) as the main components and glutaraldehyde (GA) as the cross-linking agent. The swelling ratio (expressed as a percentage) and ion-exchange capacity (IEC, measured in milliequivalents per gram) of PVA/BPEI were determined to be 99.25% and 3.01 meq/g, respectively [24].

The significance of efficient water resource management is growing, necessitating the implementation of a comprehensive system that encompasses an efficient irrigation infrastructure, minimizes water wastage, and incorporates suitable agricultural practices. Naturally occurring chemicals and heavy metals are introduced in significant quantities into several environmental compartments by anthropogenic activity. As a consequence, the capacity of the environment to support life is diminished, posing a threat to the well-being of humans, animals, and plants [25]. Superabsorbent polymers(SAP) demonstrate a high capacity to absorb water and other chemicals due to their cross-linked architectures, which render them insoluble in water. The utilization of SAP in membrane and agricultural contexts holds significant significance [26]. Furthermore, these materials have applications in the sectors of bioengineering, medicine, and cosmetics. The escalating utilization of radioactive elements contributes to the escalation of radioactive pollution, necessitating the development of modern materials for the purpose of safeguarding humans. Scholars have extensively investigated compounds that provide shielding against irradiation, namely those based on polymers (such as gamma and electromagnetic radiation) [27, 28].

The process of synthesizing colloids using lasers involves the combination of light and nanotechnology, resulting in an increased production yield of nanomaterials throughout the synthesis process. Additionally, this method has the advantage of reducing the cost per unit of laser power required for the synthesis, as stated in Ref. [29]. The utilization of laser ablation in liquids (LAL) has been identified as a viable approach for the production of diverse nanostructures in an efficient and scalable manner [30].

2. Ion exchanger

The distinctive characteristics of ion exchangers can be elucidated by their structural composition, which, as per the prevailing definition, consists of a framework that possesses an excess positive or negative charge, offset by counterions of opposite polarity. Due to the high mobility of counterions, they exhibit a propensity to be readily substituted by ions of equivalent charge. The ion-exchange capacity of an ion exchanger refers to the quantity of counterions present in its content. Selectivity is a significant attribute of ion exchangers, often referred to as the capacity to differentiate between various counter-ion species [31]. Ion-exchange resins are widely utilized in the nuclear industry for the purpose of eliminating radioactive impurities, including neutron activation products and fission products, that may have been released from fuel elements [32].

In their study, Savaskan et al. [33] made block copolymers of polystyrene and poly(ethylene glycol) (PEG) by copolymerizing styrene with two different initiators: PEG-dimethacrylate (PEG-DM) and macromonomer initiators (MIM). The PEG-DM exhibited PEG values of 400, 600, 1000, 1500, 3000, 10,000, and 35,000, while the MIM had PEG values of 400 and 1500. The extent of swelling in water (H_2O) or chloroform ($CHCl_3$) of both the sulfonated and unsulfonated block copolymers was assessed using similar experimental conditions, revealing significant variations. The investigation focused on examining the ion-exchange capacity and selectivity coefficients of the ion-exchange resins. The ion exchangers that were acquired exhibited a range of capacities, spanning from 0.4 to 2.9 meq/g. The synthesis of polymer-based ion exchangers, also known as ion-exchange resins, was initially conducted in the 1930s [34]. The cross-linked PS-PEG ion exchanger is shown in **Figure 1**.

The phenomenon of ion exchange takes place when there is interaction between an ion exchanger and a solution. The swelling ratios of ion exchangers were observed to decrease as the degree of cross-linkage increased. In the presence of an extensive quantity of ion-exchanger resins, the system would reach a state of equilibrium. At this equilibrium, the system's free energy would decrease due to the inclusion of water through the process of diluting the internal solution. This dilution would result in the release of free energy associated with mixing and a subsequent reduction in the electrostatic repulsion between adjacent fixed ions. The limitations of this process arise from the finite elasticity of the resin, which is constrained by the cross-linking mechanism. The majority of ion-exchange resins were synthesized using cross-linked copolymers of styrene and divinylbenzene, which were shaped into beads. This study pertains to the synthesis of ion exchangers using styrene PEG-DM-400, PEG-DM-600, PEG-DM-1000, PEG-DM-1500, PEG-DM-3000, PEG-DM-10,000, and PEG-DM-35,000 as starting materials. The researchers endeavored to synthesize cation-exchange resins containing PEG units through the polymerization of styrene with PEG-DMs or MIMs, followed by the sulfonation process of the resulting cross-linked polymers. In order to examine the impact of polymerization time on the degree

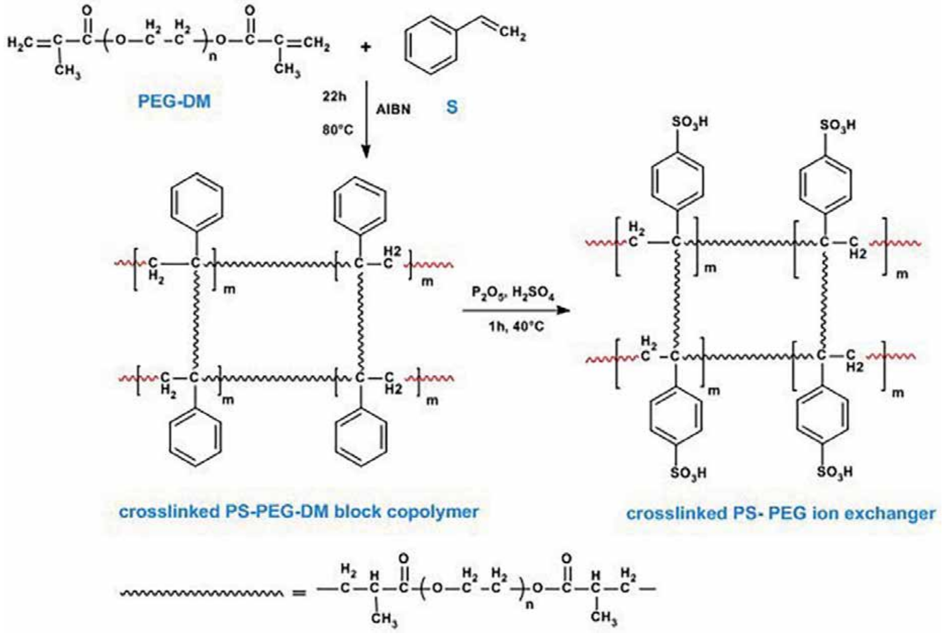

Figure 1.
The cross-linked PS-PEG ion exchanger.

of cross-linkage and ion-exchange capacity, the block copolymerization reaction was conducted for a duration of 3 and 22 hours. In order to achieve the desired objective, the process of polymerizing cross-linked poly (styrene-b-PEG) was conducted following the procedure outlined earlier. The swelling ratios observed in water cross-linked polymers were found to be significant, ranging from 48 to 313 weight percent. In terms of comparison, it was observed that the swelling ratios of the resins subjected to a shorter polymerization time (e.g., 3 hours) exhibited higher values compared to those subjected to a longer polymerization time (22 hours). The ion-exchange resin's capacity is contingent upon various factors, including the duration of the reaction, the molecular weight of the macro-crosslinkers, and the degree of cross-linkage. When the polymerization time was 22 hours, the experimental findings indicated that ion-exchanger resins exhibited a reduced ion-exchange capacity. In particular, the ion-exchange capacity of the ion-exchanger resins decreased as the polymerization time, degree of cross-linking, and molecular weight of the crosslinker all went up. The presence of polar sulfone groups in resins contributes to a higher swelling ratio of cation exchangers in water compared to the untreated cross-linked block copolymer. In the final column, the calculated capacities exhibit higher values compared to the corresponding experimental capacities. In this manner, the sulfonation of the inner core of the resin does not have an impact on the ion-exchange reaction.

Eui-Soung Jang and his colleagues [35] did a study to find out how the amount of water in polymers affects the rate of diffusion of alkali metal chlorides (specifically LiCl, NaCl, and KCl) in cross-linked poly(ethylene glycol) diacrylate (XLPEGDA) hydrogels. This study is a component of a wider endeavor aimed at comprehending the fundamental mechanisms underlying water and ion transportation in hydrated polymers. The determination of salt diffusion coefficients (Ds) was achieved through the utilization of salt permeability and sorption measurements. Hydrogels composed of XLPEGDA were synthesized utilizing three distinct water contents, specifically 50, 67, and 93 grams of water per gram of dry polymer. The Ds of alkali metal chlorides within XLPEGDA polymers exhibited an upward trend with the increase in water content. The utilization of the Mackie and Meares model, which incorporates the influence of polymer chain obstruction (also known as tortuosity) on the diffusion of salt in hydrated polymers, was employed in the analysis of salt Ds. The observed salt diffusion coefficients in these polymers align with the theoretical model, indicating a general trend. The determination of individual ion diffusion coefficients in the samples was achieved by integrating data on salt permeability/sorption with results from ionic conductivity measurements. The Ds of chloride ions exhibited uniformity across all salts. The sequence of alkali metal ion Ds observed in these polymers differed from that observed in aqueous solution, specifically with Ds K^+ > Ds Na^+ > Ds Li^+. Furthermore, it should be noted that the aforementioned order is subject to variation depending on the water content of the polymer. This observation implies that the diffusion behavior of alkali metal chlorides in XLPEGDA polymers is affected by both the hydration of ions and the specific interactions between the polymer and ions.

The present investigation selected alkali metal chlorides, such as LiCl, NaCl, and KCl, as the subject of study in order to examine the impact of ion charge density and size on diffusion within hydrated polymers. The utilization of XLPEGDA is employed as a representative polymer system in this study. The polymer in question lacks substantial electrostatic interactions between ions and polymers, allowing for a methodical examination of the influence of polymer water content on ion diffusion without substantial modification to the polymer's fundamental chemical composition. Samples of XLPEGDA with three distinct water uptake values were produced by altering the initial water content during the pre-polymerization stage. The calculation of salt Ds was performed by utilizing salt permeability and sorption data, employing the solution-diffusion model. The Ds were compared to a model proposed by Mackie and Meares, which takes into consideration the impact of polymer chain obstruction, also known as tortuosity, on the diffusion of salt in hydrated polymers. The measurement of ionic conductivity in polymer samples was conducted through the utilization of electrochemical impedance spectroscopy (EIS). This technique was then coupled with salt permeability/sorption data in order to determine the individual ion Ds for each polymer sample.

According to Feng et al. [36], sulfonated materials have been widely used in a variety of membrane applications because of their noteworthy qualities such as improved electrical conductivity, admirable chemical stability, hydrophilic nature, and decreased susceptibility to fouling. The presence of fixed charged groups and the hydrophilic nature of sulfonated polymers not only confer enhanced physicochemical properties suitable for membrane applications, but also induce notable alterations in the process of membrane formation. PEG has been extensively employed as an additive for the production of membranes from polymer solutions using the non-solvent induced phase separation (NIPS) technique. The present study aims to investigate and compare the effects of sulfonated polyphenylenesulfone (sPPSU) solutions with those of polyphenylenesulfone (PPSU) solutions, considering various thermodynamic and kinetic factors. The study revealed several key findings: Firstly, it was observed that PEG exhibited hydrogen bonding interactions with sPPSU. Secondly, the introduction of PEG into sPPSU solutions resulted in an increase in viscosity due to enhanced polymer interaction and entanglement. Lastly, the presence of PEG had a retarding effect on phase inversion and inhibited the formation of macrovoids. Furthermore, when an optimal concentration of PEG is incorporated into the dope solution, the resulting membrane exhibits enhanced mechanical integrity, increased hydrophilicity, and improved permeability characteristics. To the best of our knowledge, this study represents the inaugural investigation into the functions of PEG in the production of asymmetric membranes derived from sulfonated polymers. This study has the potential to elucidate the molecular interactions between PEG and syndiotactic polyphenylsulfone (sPPSU), thereby offering valuable insights for the development of membrane fabrication protocols.

The continuous advancement of polymeric membranes for industrial applications has been a subject of research and development for several decades. Sulfonated polymers have garnered significant interest in recent years due to their charge characteristics, which impart membranes with exceptional physicochemical properties that are well-suited for a diverse array of applications [37–40]. Previous studies have demonstrated that membranes derived from ultrafiltration and forward osmosis exhibit enhanced resistance to fouling and improved permeation properties, making them highly suitable for applications in water purification [41–43]. According to a study [44], the inclusion of sulfonic acid groups in the nanofiltration process resulted in enhanced rejections of ionic solutes. Through precise manipulation of the fabrication process, the sulfonated polymers can be customized to suit a wide range of applications and processes.

In their study, Kamcev and colleagues [45] carry out a comprehensive examination of the impact of fixed charge group concentration on the diffusion coefficients of salts in ion-exchange membranes. Cation- and anion-exchange membranes (CEMs and AEMs) with varying concentrations of fixed charge groups and comparable levels of water content were successfully synthesized using a one-step free radical copolymerization reaction. The investigation of ion transport through membranes, driven by concentration gradients, involved the measurement of salt permeability coefficients. These coefficients were measured as a function of salt concentration in the upstream solution.

The salt permeability coefficients of all membranes exhibited a notable increase of approximately tenfold as the salt concentration in the external solution increased from 0.01 to 1 M. This increase can be primarily attributed to similar enhancements in salt partition coefficients. The salt permeability coefficients for both series of membranes exhibited a decrease in average values as the concentration of fixed charge groups increased with a similar magnitude of reduction. The apparent salt diffusion coefficients, obtained from the salt permeability and salt partition coefficients using the solution-diffusion model, exhibited a more pronounced variation for the AEMs compared to the CEMs despite similar alterations in the concentration of fixed charge groups within the membranes. The observed variations in apparent salt diffusion coefficients between AEMs and CEMs were ascribed to disparities in the membranes' free volume. The hypothesis put forth in this study received support from measurements conducted using positron annihilation lifetime spectroscopy. These measurements revealed differences in the lifetime values of ortho-positronium, which serves as an indicator of the size of free volume elements within the membranes. These variations were found to be correlated with differences in the apparent diffusion coefficients of salt.

In a study by Qitao Hu et al. [46], it was discovered that the hydrophobic interaction between ions in a complex analyte could make it challenging to detect ions utilizing membrane-based ion-selective sensors. This study showcases the potential of mitigating interference caused by hydrophobic interactions on sensors through the integration of hydrophilic PEG into the membrane. The sensor employed in this study is a silicon nanowire field-effect transistor (SiNWFET) that has been modified on its surface with a mixed-matrix membrane (MMM) containing an ionophore. The ionophore used in the MMM can be either a commercially available Na-ionophore III or a newly developed synthetic metal-organic supercontainer. The inclusion of PEG hinders the distribution of hydrophobic ions within the MMM, thereby diminishing their impact on the identification of desired ions. This is supported by empirical evidence demonstrating a significant enhancement in the ability to selectively detect Na^+ ions in the presence of interfering methylene blue (MB^+) ions, surpassing a tenfold increase in selectivity. The utilization of a SiNWFET sensor array allows for the detection of Na^+ and MB^+ in a multiplexed fashion while maintaining controlled susceptibility to cross-interference and significantly expanding the dynamic range. The detection of ions in liquid samples, such as river water, sweat, and serum, holds significant importance in the fields of environmental monitoring, disease diagnosis, and medical analysis [47–50]. The utilization of sensors integrated on a single chip enables the simultaneous detection of multiple target ions, facilitating the acquisition of comprehensive and complementary information regarding the analyte [50–52]. Nevertheless, it is important to note that in practical applications, liquid samples typically consist of intricate constituents, including elemental ions, molecular ions, and biomolecules. The presence of intricate components has the potential to disrupt the functionality of ion sensors, leading to the generation of inaccurate and irrelevant responses. This interference can result in selectivity issues [52–54].

Kulshrestha and colleagues [55] present in their study a comprehensive analysis of the extensive production and characterization of interpolymer ion-exchange membranes (IEMs) based on PE. Furthermore, they assess the performance of these membranes in the context of water desalination through electrodialysis. The CEM was fabricated using a cross-linked film made of PE and polystyrene interpolymers. Similarly, the AEM was prepared using a cross-linked film made of PE and polypmethylstyrene interpolymer through an appropriate functionalization reaction. Both the prepared CEM and AEM demonstrated an ion-exchange capacity of 1.5–1.30 meq g^{-1}, ionic conductivity ranging from 2.85–1.15 mS cm^{-1}, and transport numbers of 0.92–0.93, respectively. The AEM and CEM were employed in the desalination of brackish water with a total solid content ranging from 2000 to 3000 mg/L. This process was carried out using a unit with a size of 200 cm^2, utilizing 30 membranes of each type. The desalination was conducted in a single-pass mode, resulting in a final flow rate of purified water ranging from 7.2 to 8.4 L/h. The desalination processes exhibited power consumption values ranging from 0.789 to 0.796 kWh/kg, while the current efficiency ranged from 87 to 86%. In contrast, the W and CE% values observed for the commercial membrane (IONSEP) during water desalination under similar experimental conditions were found to be 1.125–1.07 kWh/kg and 61–64%, respectively. The aforementioned values demonstrate the superior performance of the developed membranes and confirm the successful implementation of the process on a large scale. **Table 1** presents the ion-exchange capacity (meq/g) and swelling ratios (%) of ion-exchange resins and membranes that incorporate polyethylene glycol and polyethylene.

Sample	Ion-exchange capacity $_{exp}$ (meq g^{-1})	Swelling ratio (%)
R-DM-400 [33]	2.82	311.00
R-DM-600 [33]	2.04	300.00
R-DM-1000 [33]	0.55	133.46
R-DM-1500 [33]	1.81	280.80
R-DM-3000 [33]	0.30	399.23
R-DM-35000 [33]	1.00	467.54
CA200 [45]	2.04	—
CA238 [45]	2.48	—
CEM-1 [55]	1.43	—
AEM-1 [55]	1.00	—
PE-g-PVBC-TOH (8.3%) [56]	0.40	9.46
PE-g-PVBC-TOH (12.6%) [56]	0.49	14.67
PE-g-PVBC-TOH (15.7%) [56]	0.58	22.53
PE-g-PVBC-TOH (17.4%) [56]	0.63	23.05
PE/PSt [57]	1.32	—
PE/P4-MS [57]	1.30	—
Ionsep [57]	2.20	
Nafions [58]	0.84	16
Neoseptas [58]	1.78	17
CMX MSC-0-23 [58]	1.60	26
MSC-0-31 [58]	1.98	25
MSC-1.5-27 [58]	1.73	23
MSC-3.5-29 MSC-3.5-32 [58]	1.57	22
MSC-3.5-32 [58]	1.65	20

Table 1.
The ion-exchange capacity and swelling ratios of some ion-exchange resins and membranes.

Ion-exchange membranes can be categorized into two main types: CEMs and AEMs, based on the specific functional group that is attached to the surface of the membrane [59–62]. The inter-polymer membrane, which is composed of PE and polystyrene, exhibits several advantageous characteristics. Firstly, it possesses a morphology wherein the ionic phase is dispersed within a hydrophobic matrix. Additionally, this membrane is cost-effective and can be easily processed. The AEMs fabricated utilizing CME exhibit high efficiency across various applications. Nevertheless, in light of the potential harm to human health caused by this process, alternative methods have been utilized for the preparation of AEM. A novel AEM based on the inter-polymerization of PE and polyp-methylstyrene (P-MS) through benzylic bromination was synthesized and employed for the purpose of water desalination [34]. This study showcases the extensive production of CEM and AEM through the utilization of inter-polymer films composed of PE and polystyrene (PSt) for CEM and PE and poly(methyl methacrylate-co-styrene) (PMS) for AEM. These membranes were subsequently employed in the process of electrodialysis (ED) for the desalination of brackish water. The desalination efficiency of these membranes has been evaluated in comparison with the commercially available membrane (IONSEP), as well as the AEM based on PE/PSt interpolymer prepared through conventional means using CME. Research has indicated that the efficiency of a PE/PMS membrane is similar to that of a PE/PSt membrane.

In their work, Mourad Amara et al. [63] examined the utilization of commercial polymer cation-exchange resins for the purpose of separating and recovering Cu(II) and Ag(I) from synthetic solutions. After altering the properties of the cation-exchange resin through the fixation of organic macro-cations under different experimental conditions, these substances exhibit a range of ionic species. The modification process involved impregnating the resin under varying conditions, such as immersion time, concentration, and pH. This was done by immersing the resin into solutions containing tetramethylammonium, tetrabutylammonium, and polyethyleneimine. The determination of the selectivity coefficient and resin capacity for the ions Li(I), K(I), Fe(III), Cu(II), Ag(O), and Cd(II) was conducted in batch operations. Additionally, the kinetics of metal adsorption by both the modified and unmodified resins was investigated. Following the adsorption of the organic macro-cation, the ion-exchange capacity exhibited variability, resulting in a decrease in the rate of the ion-exchange reaction for monovalent ions. However, the modification of the cation-exchange resin in a polyethyleneimine solution led to an increase in the exchange capacity specifically for copper ions. In the conducted batch experiments, various stripping solutions were evaluated for their effectiveness in recovering metals that had been loaded onto the resin during the adsorption operation. These solutions included SCN 0.01 M, HNO_3 0.5 M, HNO_3 1 M, and thiourea 0.1 M in HNO_3 0.5 M. The obtained results were as follows when the tested solutions were composed of a mixture of ions adsorbed in the resin. The elution efficiency of Cu(II) immobilized on PEI-modified resin using acids exhibited a 30% decrease in comparison with the 65% elution efficiency observed with unmodified resin. In contrast, it was found that a solution containing thiourea at a concentration of 0.1 M in nitric acid at a concentration of 0.5 M exhibited the highest efficacy in eluting both silver ions (Ag(I)) and copper ions (Cu(II)), resulting in an equal recovery rate of 80% for each metal.

The physicochemical properties of experimental heterogeneous MK-40 and MA-41 membranes, composed of varying ratios of ion-exchange resin and PE binder, were investigated by V.I. Vasilieva et al. [64] in their study. The data demonstrates that the specific electrical conductivity in NaCl solutions ranging from 0.01 to

1 M exhibits a threefold decrease for cation-exchange membranes and a twofold decrease for AEM as the ion-exchange resin content in the membranes decreases from 69 to 55%. The sensitivity of diffusion permeability in AEM to their composition has been established, whereby an increase in the proportion of ion-exchange resin in the composition naturally leads to an increase in permeability. The impact of the diverse composition of a membrane on its structure, as determined through standard contact porosimetry, is further enhanced by calculating the transport-structural parameters of both the micro-heterogeneous and extended three-wire models of the ion-exchange membrane. The investigation into the interplay between composition, structure, and properties is a fundamental inquiry within the field of membrane materials science [65]. Previous research has investigated the impact of the ion-exchange resin proportion in CEMs on the advancement of electroconvection. However, it is important to note that these studies were conducted using samples of Ralex CM (MEGA a.s.) from foreign sources.

The study conducted in Refs. [66, 67] examined the impact of the PE to polystyrene sulfonic cation-exchange resin ratio on the conductive properties of the MK-40 membrane, which is manufactured by Shchekinoazot in Russia. In the practical application of high-intensity electrodialysis and electrodialysis preconcentration of electrolyte solutions, it is crucial to determine the acceptable ranges of variation in the ratio between the ion-exchange resin and the inert binder. These ranges should ensure that there are no substantial alterations in both the specific electrical conductivity and the diffusion permeability of the membranes. No investigations have been conducted on heterogeneous CEM and AEM. Therefore, the investigation of the structural and electro-transport properties of heterogeneous membranes containing varying proportions of ion-exchange resin and inert polymer remains pertinent. The objective of this study was to investigate the impact of the composition of heterogeneous membranes MK-40 and MA-41 on their structural and electro-transport properties. The objective of this study was to conduct an experimental investigation on the electrical conductivity, diffusion permeability, and water distribution across different binding energies and pore radii in heterogeneous membranes. These membranes were composed of varying amounts of resin and PE. Additionally, the transport and structural parameters of the membranes were calculated using the two-phase and extended three-wire models of ion-exchange material conductivity. The aim was to gain new insights into the influence of membrane composition on their structure and properties, and to evaluate their potential applications in electromembrane processes.

In their study, Naim et al. [68] discuss the preparation of heterogeneous membranes through the incorporation of ion-exchange (IE) resins, specifically cation or anion resins, into a polymer matrix. This integration ensures that the resin is uniformly distributed within the polymer, resulting in a homogeneous structure. This study focuses on the fabrication of new membranes and the investigation of various variables. These variables include the type of polymer matrix used, specifically cellulose acetate (CA) or cellulose acetate butyrate (CAB), as well as the source and properties of the CA. Additionally, the study examines de-esterified membranes, different types of ion-exchange (IE) resins (cationic, anionic, or nonionic), and the use of either ground or pristine resins. The experimental results revealed that the utilization of ground resins led to the production of thinner membranes that exhibited enhanced perm-selectivity for both cationic and anionic ion-exchange resins, using the two tested brands of cellulose acetate. The nonionic ion-exchange resin yielded anticipated unfavorable outcomes, particularly when it underwent pulverization prior to its amalgamation with the polymers. The results obtained from the CAB polymer were found to be unfavorable in comparison with the two types of CA polymers investigated. Additionally, the CAB polymer exhibited lower perm-selectivity, particularly when the IE resins were ground prior to their incorporation into the CAB polymer. Bouzek et al. [69] prepared ion-exchange membranes with heterogeneous characteristics by blending sulfonated poly(1,4-phenylene sulfide) or sulfonated styrene–divinylbenzene copolymer particles with a matrix polymer. A total of four different types of polymers were utilized in this study as a matrix. These polymers included highly flexible linear PE, moderately flexible fluoroelastomer, rigid polystyrene (all of which exhibit high hydrophobicity), and hydrophilic cellulose that was prepared through the process of hydrolysis of CAB. The investigation of membrane morphologies was conducted using scanning electron microscopy (SEM), infrared spectroscopy (IR), and density measurements.

3. Superabsorbent polymers

SAPs are a class of polymeric materials that exhibit exceptional water absorption properties. Superabsorbent materials, known as “slush powder” in the scientific literature, are composed of a complex three-dimensional structure of hydrophilic polymers that are cross-linked. These polymers, which can be linear or branched, possess the ability to absorb and retain a wide range of fluids, including bodily fluids and blood solutions [70]. The master’s project submitted to the patent by the research group led by Savaskan Yilmaz involved the synthesis of the SAP and the ion-imprinted polymers (IIP) using AA and PEG monomers [71]. The present study investigated the adsorption rate, adsorption capacity, and desorption rate of heavy metal ions, including Mn^{2+}, Ni^{2+}, Cu^{2+}, Zn^{2+}, Pb^{2+}, Cd^{2+}, Cr^{3+}, Co^{2+}, and Fe^{3+}, using PEG SAPs. The observed adsorption capacity of heavy metal ions ranged from 31.52 to 396 mg/g. The removal of metal ions from the SAPs was achieved with remarkably high efficiency, reaching up to 97%. The maximum water absorbency of the SAPs at pH = 9 was found to be 2585.3 weight percent (**Figure 2**) [71].

Superabsorbent hydrogels, characterized by their water-soluble structure, can be transformed into insoluble forms through various cross-linking techniques. Hence, these substances can be classified as hydrophilic polymers, exhibiting insolubility in water while demonstrating notable water absorption capabilities. The performance of absorption capacity is directly influenced by the densities of cross-linking and charge in superabsorbent materials [72]. Due to their porous nature, they are frequently favored for the purpose of retaining heavy metal ions. Due to its notable swelling capacity and user-friendly characteristics, this material finds applications in various fields, including biomedicine [73], drug delivery [74], personal care products [75], batteries and tissue engineering, construction industry [76], food packaging [77], electronics and cables [78], cosmetics [79], and agriculture [80]. These diverse applications highlight the widespread utilization of this material.

Figure 2.
SEM image of PEG doped superabsorbent polymer and its swelling in water over time [71].

In recent times, various hydrogel materials, including poly(acrylic acid), poly(acrylic acid) grafted cellulose, and macroporous poly(glycidyl methacrylate-co-ethylene glycol dimethacrylate), have been documented as effective agents for the elimination of Cu(II), Cd(II), and Cr(III) in scenarios involving competitive conditions. In their study, Tang et al. successfully produced a hydrogel with an interlocking network (IPN) structure by means of a straightforward two-step aqueous solution polymerization process. The hydrogel was synthesized using a combination of polyacrylate and PEG (PAC/PEG) materials (**Figure 3**).

In the first step of the polymerization process, acrylic acid (AA) and acrylate (AC) monomers are combined to make a prepolymer called PAC. Subsequently, a copolymerization reaction occurs in step II between the PAC prepolymer and PEG, resulting in the formation of an interpenetrating polymer network (IPN) structure. All the above materials were purchased from Shanghai Chemical Reagents Co., China. The hydrogel that was obtained demonstrated a greater capacity for adsorption when the degree of neutralization was increased, the dosage of AA was increased, and the temperature was decreased. The experimental findings indicated that the quantity of heavy-metal ions adsorbed during the deswelling process was greater in comparison with the adsorption observed during the swelling process [81].

SAPs find extensive application in the production of agricultural fertilizers and wastewater treatment processes. This is primarily attributed to the effective interaction between hydrophilic groups present in the polymer network and specific pollutants such as ammonium nitrogen, heavy metals, and various dyes [82]. Nevertheless, traditional SAPs predominantly consist of synthetic polymers derived from petroleum, which exhibit a high manufacturing expense and limited biodegradability. Copolymers typically consist of anionic and nonionic monomers, with acrylic acid and acrylamide being particularly common examples. Consequently, the formulated SAPs exhibit a predominantly polyanionic nature, thereby frequently falling short of fulfilling the demands imposed by intricate circumstances owing to the uniformity of the ionic composition [83].

In the present context, the synthesis of amphoteric SAPs incorporating hydrophilic groups with both positive and negative charges has emerged as a viable and promising approach for producing SAP. This development has garnered significant attention from numerous researchers. According to Sanjuan and Tran [84], the material demonstrates a net charge that can be either negative, positive, or neutral. Zhao and Su [85] have observed that the material also possesses a distinctive reversible stimulation response. Additionally, Xu [86] has found that the material exhibits a counter-ion extrusion effect and displays superior salt tolerance compared to monoionic polyelectrolytes.

The characteristic features of amphoteric SAPs that contribute to their wide range of applications include the ratio of opposite charges, the degree of dissociation of counterions, intramolecular and intermolecular hydrogen bonding, and coulombic interactions between opposite charges in the network structure. These features have made

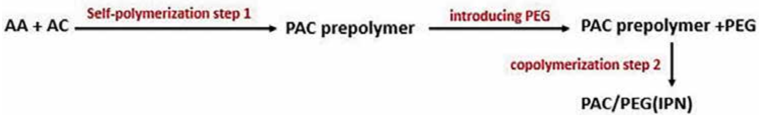

Figure 3.
Super absorbent hydrogel synthesized from polyacrylate and polyethylene glycol (PAC/PEG) materials.

amphoteric SAPs highly desirable in various fields such as agriculture and horticulture, selective adsorption, bio-separation, fertilizer coatings, controlled drug release, smart materials for biomedical devices, and cardiovascular disease treatment [87].

Waste plastics, composed of diverse synthetic polymers, possess the capacity to undergo chemical modifications and functionalization processes, resulting in the formation of SAPs. Despite the commendable characteristics and extensive range of applications associated with plastics, a substantial quantity of plastic waste is generated annually, constituting approximately 10.6 ± 5.1 weight percent of municipal solid waste [88].

According to Zhou and Fang [89], PE is a predominant constituent of municipal plastic, accounting for approximately 69% of total plastic waste. Due to their nondegradable nature, waste plastics have the potential to cause significant adverse effects on both human and animal populations. The predominant method of disposal for these materials involves landfilling and incineration, often without adhering to appropriate waste management practices. In contrast, limited attention has been given to the functionalization of waste plastics in existing reports. Waste plastics possess the potential for modification *via* hydrogen extraction, enabling their utilization as skeletal materials for chemical reactions with hydrophilic monomers. This process involves the presence of an initiator and crosslinker, facilitating the preparation of SAPs. In contrast to other recycling methods such as incineration, mechanical recycling, and chemical recycling [88], the production of SAPs offers numerous technological and economic benefits. These include the utilization of mild reaction conditions, simplified operational procedures, cost-effectiveness, and the generation of high-value products. The utilization of plastic waste for the production of SAPs presents a viable and advantageous alternative for the management of plastic waste, owing to its notable economic and ecological advantages.

In their study, Zhang et al. [90] conducted research with the objective of producing SAP by utilizing waste PE film. Initially, a mass of 1.00 g of waste PEG film (which main component is linear low-density polyethylene (LLDPE), which was provided by Shanghai CLEANWRAP plastic Co. Ltd. Shanghai, China) is fully dissolved in a specific quantity of toluene (analytical grade) at a temperature of 93°C. Prior to the reaction, the reactor undergoes a nitrogen purging process in order to eliminate oxygen from the system. Subsequently, different concentrations of benzoyl peroxide (BPO) dissolved in toluene are introduced into the mixture as an initiator, thereby generating radicals. Monomers comprising dimethyl diallyl ammonium chloride (DMDAAC) and partially neutralized acrylic acid (AA) at a mass ratio ranging from 1:16 to 3:16 (m/m) are incrementally introduced into the solution. The density of these monomers falls within the range of 0.16 to 0.19 g/mL. In order to determine the ultimate water/oil ratio, a specific quantity of MBA (N,N′-methylenebisacrylamide) solution is introduced and thoroughly blended for a duration of 25 minutes, resulting in the formation of an emulsion. Ultimately, the resulting products undergo multiple rinses with alcohol and distilled water in order to eliminate any residual monomers and acrylic acid homopolymers. The chemical reagents used in this study were DMDAAC, a 60 weight percent aqueous solution, span 60, and benzoyl peroxide (BPO), all obtained from Sinopharm Chemical Reagent Co. Ltd. In Beijing, China. The acrylic acid (AA) used in this study was obtained from Tianjin Kemiou Chemical Reagent Co. Ltd., located in Tianjin, China. The supplied acrylic acid was chemically pure. N,N′-methylenebisacrylamide (MBA), obtained from the Tianjin Fuchen Chemical Reagent Factory in Tianjin, China, was dissolved in distilled water prior to its utilization.

The quantity of crosslinker is a crucial factor in the development of a highly efficient three-dimensional network structure during the polymerization process. Theoretical studies have indicated that the amount of crosslinker is inversely related to the water absorption capacity of SAP. In this study, a series of innovative amphoteric SAR PE films, consisting of grafted glycidyl methacrylate (g-AA) and dimethyl diallyl ammonium chloride (DMDAAC), were effectively synthesized through reverse emulsion polymerization using waste materials. The amphoteric superabsorbent resins (SARs) exhibited maximum water absorption capacities of 286.3 g/g in distilled water and 213.5 g/g in rainwater. Furthermore, the polymer that was prepared demonstrated a distinct poly amphoteric characteristic across a broad pH spectrum and exhibited the ability to be utilized repeatedly. This research paper introduces a novel, intermediate, and user-friendly methodology for the recycling of plastic waste. Moreover, due to their exceptional properties, the products hold significant potential for utilization in various fields such as agriculture, horticulture, wastewater treatment, civil works, smart materials, and sponge city construction [90].

The majority of prevalent and commercially available superabsorbent materials currently utilized are derived from acrylic acid polymers and copolymers, which are sourced from petroleum and possess the characteristics of being nonrenewable and nonbiodegradable. The escalating apprehensions regarding sustainability and the enduring accessibility of petrochemical feedstocks have generated a growing fascination and potential for the production of superabsorbent materials derived from renewable sources. The integration of bio-based superabsorbent hydrogel production aligns effectively with the biorefinery concept, facilitating the comprehensive utilization of lignocellulosic biomass for the production of biofuels and value-added products. Furthermore, it should be noted that superabsorbent materials derived from natural polymers possess the desirable characteristics of being biodegradable, nontoxic, and biocompatible [70].

In order to address these constraints, researchers have investigated the use of natural polymers, including polysaccharides such as starch, cellulose, chitosan, carrageenan, alginate, and gum arabic, as well as proteins and gelatin cross-linked synthetic polymers, as potential substitutes for petroleum-derived synthetic SAPs.

Choi and colleagues [91] recently published a study introducing a novel sustainable SAP based on poly(itaconic acid) (PIA). Itaconic acid (IA) is an organic acid that is synthesized by the fungus Aspergillus terreus. It is derived from the thermal decomposition of citric acid and possesses a hydrophilic functional group. IA, a bio-derived fermentation feedstock, finds extensive utilization in various industrial sectors such as the manufacturing of synthetic resins, synthetic fibers, plastics, rubbers, surfactants, and oil additives. This is primarily due to its favorable characteristics of being biodegradable and nontoxic. PIA is synthesized through a radial polymerization process, employing IA as the sole monomer. The aforementioned procedure involves the formation of double bonds with IA through the utilization of potassium persulfate (KPS) as an initiator. IA (>99%) monomer and KPS (95%) were purchased from Samchun Pure Chemistry Co., Ltd. (Pyeongtaek, Korea).

The cross-linked PIA superabsorbent (c-PIAS) was then prepared by adding different amounts of PEG diacrylate (PEGDA) as the cross-linking agent to the water-soluble PIA. PEGDA (MW 700 Da) was purchased from Sigma-Aldrich Corporation (St. Louis).

In brief, the production of bio-based c-PIAS was effectively achieved by employing IA as the monomer and PEGDA as the cross-linking agent. The morphology of c-PIAS exhibited two distinct forms, namely a porous mixed structure and a honeycomb structure. According to Choi and Park [91], certain c-PIAS samples that possess a mixed structure exhibit a notable capacity for water absorption. Conversely, c-PIAS samples with a honeycomb structure demonstrate a significant absorption capability when subjected to pressure.

4. Radiation shielding

Radiation shields have been of utmost importance in safeguarding against ionizing radiation in various industrial and medical contexts [92, 93]. The failure to adhere to radiation protection measures poses a significant risk to individuals as exposure to ionizing radiation is considered a highly perilous threat [94]. This risk is applicable regardless of whether the source of radiation stems from the extraction of materials during mining and oil extraction activities or their subsequent transportation and storage. The growing advancements in mining and oil extraction techniques have led to a heightened interest in the development of materials that can effectively prevent radiation leakage. This is crucial in order to safeguard individuals from potential exposure to ionizing radiation [95]. The fundamental and significant factors in radiation therapies and the associated advancement procedures revolve around safeguarding individuals from the detrimental consequences of neutron radiation, as well as the photons discharged by gamma rays and X-rays.

The utilization of polymeric composite materials is progressively growing as a means to mitigate radiation hazards. PE is frequently employed as a material for storing and transporting naturally occurring radioactive substances due to its high hydrogen content, which enables it to effectively absorb neutrons and function as a radiation shield. According to Reis et al. [96], HDPE is considered to be highly suitable for use in packaging and manufacturing various products. High-density HDPE exhibits favorable mechanical characteristics as a hydrocarbon-based polymer. It is cost-effective, possesses high impact resistance, is easily processed, and demonstrates compatibility with a wide range of other polymers [97].

The researchers, Khozemy et al. [98], were able to effectively produce composites that serve as radiation shielding materials. These composites were specifically designed for the purpose of managing naturally occurring radioactive waste. The composites in question were fabricated utilizing a polymer matrix composed of HDPE, which was further strengthened through the incorporation of Pb_2O_3 and $Al(OH)_3$ materials. The manufacturing procedure entailed the utilization of thermal molding methods. The composites that were prepared demonstrated adjustable attenuation coefficients, which showed a proportional increase with the $Al(OH)_3$ content present in the sheets. The incorporation of 50% aluminum hydroxide ($Al(OH)_3$) and 10% lead oxide (Pb_2O_3) into the sample results in a notable enhancement of its gamma-ray shielding capabilities. This improvement is approximately sixfold when compared to the shielding properties of pure HDPE without any reinforcing materials. The increase in the concentration of $Al(OH)_3$ and the level of irradiation dosage result in a slight improvement in the tensile strength while simultaneously causing a decrease in the elongation. Additionally, it has been observed that the inclusion of $Al(OH)_3$ composites in HDPE samples results in notable enhancements in thermal stability, thereby yielding favorable and satisfactory outcomes. The composite sheets employed in this research demonstrate cost-effectiveness and exhibit superior efficacy in reducing the impact of ionizing radiation. Moreover, these materials demonstrate significant mechanical characteristics and display advantageous thermal stability, making them suitable for the construction of large containers that are appropriate for storing and transporting naturally occurring radioactive materials.

Aldhuhaibat and colleagues [99] conducted a study to examine the properties of HDPE reinforced composite shields. These shields were enhanced with different metal oxides to assess their effectiveness in attenuating γ-ray radiation, as measured by parameters such as linear attenuation coefficient (μ), mean free path (MFP), half-value layer (HVL), and transmission factor (TF). The researchers discovered that shields coated with PbO demonstrate enhanced attenuation capacity and efficiency in comparison with shields composed of Al_2O_3 and Fe_2O_3. Moreover, the attenuation capacity of the shield is further enhanced by increasing the PbO content. The composite shield consisting of 50% PbO and HDPE demonstrated superior performance across all investigated γ-ray energies, exhibiting the highest value for μ (linear attenuation coefficient) and the smallest values for MFP, HVL (half-value layer), and TF (transmission factor). The composite shield consisting of 50% PbO and HDPE demonstrated superior performance across all investigated γ-ray energies, exhibiting the highest value for the linear attenuation coefficient (μ) and the smallest values for the MFP, HVL, and TVL. The investigation of the relationship between shield material density and various shield types, including pure HDPE and composite shields with varying concentrations of lead oxide (PbO), demonstrated a noticeable increase in the values of μ (a measure of shielding effectiveness). Conversely, the values of MFP, HVL, and TF exhibited a decrease. At the minimum energy level of γ-rays, which is 0.295 MeV, the values of μ are maximized, whereas the values of MFP, HVL, and TVL are minimized. In this study, it was observed that HDPE shields fortified with 0–30% Al and Fe had the lowest values of HVL and efficiency rates of 7.7262 and 18.9230%, respectively. Additionally, it was found that a shield fortified with 0–30% PbO had the lowest HVL values. Demonstrated their core principles and achieved the utmost levels of productivity of 61.4881%. All of the calculations demonstrated a high level of concordance between the experimental and theoretical values. Furthermore, the utilization of PbO as a reinforcing agent resulted in enhancements in both Reinforcement Efficiency Factor (REF) and Displacement of Pb (dPb) calculations. Notably, this improvement was observed to be directly proportional to the concentration of the reinforcement, as observed across all experimental and theoretical energy measurements. Furthermore, it can be observed that the behavior of REF exhibits an inverse relationship to that of dPb as the energy of γ-rays increases. Specifically, the values of REF decrease exponentially, while the values of dPb increase exponentially in response to the escalating γ-ray energy. The utilization of composite shielding comprising 50% PbO/HDPE has been demonstrated as the optimal and highly efficacious method for mitigating the potential hazards associated with radiation exposure.

The study conducted by Malekie et al. [100] aimed to investigate the radiation shielding properties of a composite material composed of bismuth oxide and HDPE. The study focused specifically on the composite's effectiveness in shielding against gamma rays with an energy of 59.5 keV. To assess these properties, the researchers employed the MCNP, XCOM, and experimental methodologies. The simulation results demonstrated a positive correlation between the weight percentage of bismuth oxide in the polymer matrix and the attenuation coefficient of the composites. This finding aligns well with the data obtained from XCOM. Subsequently, a nanocomposite consisting of 60% Bi_2O_3 was synthesized using the casting technique. The dispersion state of Bi_2O_3 nanoparticles within the polymer matrix was examined using X-ray diffraction (XRD) and field emission scanning electron microscopy (FESEM) techniques. The results revealed a semicrystalline behavior, indicating the presence of agglomerations within the polymer matrix. Two techniques, namely the CsI(Tl) scintillation detector and Geiger counter, were employed to measure the attenuation coefficient of the samples against the 241 Am source at an energy of 59.5 keV. The values of μ, μ/ρ, HVL, and TVL quantities for the 60 weight percent composite were determined using various simulation and experimental techniques, demonstrating a strong correlation between the two. The outcomes of the examination conducted on the nanocomposite artifact using a dental X-ray system operating at 60 kV, while imaging an animal jaw revealed that the composite material effectively absorbed the X-rays without causing any detrimental impact on the image's quality. The findings of this study indicate that the HDPE-Bi_2O_3 nanocomposite possesses the potential to serve as an effective barrier against dental radiography photons and X-rays. It is imperative to underscore the necessity of conducting further comprehensive studies, including precise dosimetry utilizing standard phantoms, in order to employ this nanocomposite, shield for human applications in dental radiography in the future.

The prospective utilization of a polymer with high hydrogen content, combined with a filler material of low atomic mass, exhibits considerable potential as a protective substance, thus offering promising prospects for future applications. The researchers, Harrison et al. [101] demonstrated that the inclusion of boron did not yield a substantial advantage until lower levels of neutron energies were attained. The addition of BN to PE leads to enhanced mechanical characteristics in comparison with unadulterated PE. The implementation of surface modifications on BN particles yields enhanced mechanical properties in comparison to untreated powders. The inclusion of boron nitride (BN) in a composite material leads to an enhancement in the tensile modulus properties as compared to the pure form of the polymer material. The value of the powder surfaces is enhanced through the process of functionalization. The addition of BN powder exhibits negligible impact on the tensile strength in comparison with HDPE. Nevertheless, the incorporation of the coupling agent leads to a more significant enhancement in tensile strength when compared to HDPE.

PE is widely recognized as a material suitable for shielding applications due to its significant hydrogen content. Hydrogen, possessing an atomic number of one, exhibits the most substantial energy dissipation per unit mass. The mean free path of ions in hydrogen is comparatively shorter than that in other materials, resulting in increased fragmentation of heavy ions and a reduced dosage administered. The integration of boron carbide and PE in a composite material has the potential to offer enhanced shielding properties compared to pure PE. The inclusion of filler materials within a PE matrix is expected to enhance the mechanical properties of the resulting composite. A study conducted by Harrison et al. [101] investigated the mechanical properties of BN/PE composites and found that they exhibited enhanced characteristics compared to pure PE.

PE/boron carbide composites play a crucial role in space radiation shielding applications due to their ability to serve as both structural materials and radiation shields, thereby offering distinct advantages. Harrison et al. [102] conducted a study to determine the mechanical and shielding properties of both composites and neat HDPE. Low atomic mass fillers, such as boron, have been found to be advantageous in hydrogen-rich materials as shielding agents. The incorporation of boron carbide into HDPE has been observed to result in enhanced mechanical characteristics. HDPE exhibits a tensile modulus of 588 MPa, which experiences an increase to 943.1 MPa when incorporating 15 vol% of untreated boron carbide powder. These composite materials exhibited superior performance compared to other composites containing boron in various high-energy neutron and proton facilities, demonstrating their effectiveness as shielding materials. The lower density of this material provides it with an advantage over its conventional aluminum counterpart. Furthermore, the merit of PE-based materials lies in their capacity to shield radiation fields without generating long-lived or high-energy gamma-emitting offspring. Ongoing research is being conducted to evaluate the suitability of the material as a low-energy neutron shielding material for the purpose of mitigating neutron spectra within and in the vicinity of nuclear reactors.

The risk of exposure to space radiation poses a significant obstacle in the context of human space exploration. This issue becomes particularly worrisome when considering prolonged exposure in the context of extended space travel, such as during long-duration space flights, low-Earth orbit missions, and expeditions to near-Earth asteroids. In order to tackle this matter, the utilization of multifunctional materials has emerged as a prominent strategy for mitigating the effects of space radiation. Based on the latest NASA Technology Roadmaps, two prominent technology requirements within the top three technology objectives are radiation mitigation for human spaceflight and the development of lightweight and multifunctional materials and structures. The study conducted by Herrman et al. [103] centered on the examination and description of the physical, thermal, and radiation shielding attributes of thermoplastic PE/BN composites. BN composites have been documented as being utilized in applications involving radiation shielding. Nevertheless, the BN fillers employed in these composites exhibited a deficiency in interfacial adhesion with the HDPE matrix, resulting in a restricted enhancement or even deterioration of the physical and radiation shielding properties of HDPE/BN. A base material consisting of HDPE of injection-molding grade was employed. The reinforcement materials chosen for this study were boron carbide and BN, both with particle sizes smaller than 10 m. The suitability of thermoplastic formulations containing PE-BN composites was assessed as a prospective material for radiation shielding in space applications with the aim of safeguarding against galactic cosmic radiation. The utilization of atomic force microscopy (AFM) in the investigation revealed that the blends comprising PE and BN exhibited a satisfactory level of homogeneity when BN concentrations were low. However, as the BN content increased to higher proportions, the uniformity of the blends was observed to decrease. Furthermore, it was noted that the inclusion of BN at a low percentage resulted in a decrease in the compressive strength of these materials, potentially attributable to the lubricating properties of BN. The compressive strength was found to be enhanced with increased levels of BN. The results obtained from the neutron exposure experiments indicate a direct relationship between the initial activity level and the effectiveness of shielding. Specifically, it was observed that a decrease in initial activity corresponds to an increase in shielding effectiveness. Additionally, it has been demonstrated that the quantity of BN within the specimen has an impact on the mass absorption.

Di Fino et al. [104] conducted a study on the International Space Station (ISS) to evaluate the efficacy of PE and Kevlar materials. This evaluation was performed using three detectors of the ALTEA system [105–107] over a period of time spanning from June 8, 2012 to November 13, 2012. The study was conducted specifically in Express Rack 3 located in the Columbus module of the ISS. These active detectors have the capability to determine the radiation quality parameters in any orbital region. They are identical and can be used simultaneously with one detector serving as the unshielded baseline and the other two detectors measuring radiation with two different amounts of the same material (5 and 10 g/cm^2). There is a well-documented similarity in the shielding behavior exhibited by PE and Kevlar. The researchers conducted measurements on the shielding phenomenon, which resulted in a significant reduction of approximately 40% for ions with high atomic numbers. The measurements are presented as ratios relative to the baseline measurements obtained without any shielding material, within the range of 3 to 350 keV/μm for linear energy transfer (LET). The observed decreases in dosage for the 10 g/cm2 shields designed to mitigate high LET radiation (>50 keV/μm, not depicted in the diagram) align with the findings from accelerator measurements (specifically, iron ions at 1 GeV) [4]. In their measurements, it has been observed that the thinner shielding, with a density of 5 g/cm^2, exhibits approximately a 2% improvement in performance in terms of unit areal density.

Almurayshid et al. [108] conducted an empirical investigation to evaluate the efficacy of different polymer composites in the context of radiation shielding. Compared to lead (Pb), these materials possess the characteristics of being lightweight and nontoxic, rendering them potentially suitable for use as shielding agents in diagnostic radiology applications, particularly in scenarios where low-energy photons are predominantly employed. Their research paper presents an investigation into the fabrication of four composite materials by incorporating a primary component, namely HDPE polymer, with four additional constituents, namely molybdenum, molybdenum carbide, tungsten, or tungsten carbide. The composites obtained were synthesized as 20 disks, with each disk having a thickness of 2 mm. The disks underwent irradiation using a kilovoltage X-ray source with a field size of 1.5 cm^2. The measurements were conducted to determine the mass attenuation coefficient (μ_m), HVL, MFP, and equivalent atomic number of the disks. It was observed that the addition of additives to HDPE resulted in an improvement in beam attenuation. The composites exhibited higher μm values compared to the pure HDPE polymer. The micrometer (μm) values obtained from the measurements and calculations exhibited a high level of concordance, with an average discrepancy of 5.2%. The experimental investigation involved testing three different additive concentrations, specifically 5, 10, and 15%. The results indicated that the HDPE sample with a 15% concentration exhibited the highest shielding efficiency. The polymer composites containing tungsten (W) and tungsten carbide (WC) demonstrated superior performance in attenuating radiation beams due to their smaller values of half-value layer (HVL) and MFP.

The study conducted by Cinan et al. [109] examined the gamma-ray shielding properties of cross-linked PS-b-PEG when blended with nanostructured selenium dioxide (SeO_2) and BN particles. That study provides an overview of various factors related to radiation shielding, including the μ_m, linear attenuation coefficient (μL), radiation protection efficiency (RPE), HVL, TVL, and MFP. The investigation of the irradiation properties of our nanocomposites was conducted using rays emitted by a 152Eu source within the energy range of 121.780 to 1408.010 keV. That was done utilizing a high-purity germanium (HPGe) detector system, and the data obtained was analyzed using Gamma Vision software. The incorporation of PS-b-PEG copolymer, nanostructured SeO_2, and BN particles resulted in a notable enhancement of the resistance properties of the nanocomposites. Moreover, the nanocomposites with higher additive rates demonstrated superior resistance compared to the other nanocomposites. The observation of the attenuation characteristics of gamma rays in a wide energy range can be achieved by calculating the RPE rates of our polymer-based nanocomposites. The findings of our study demonstrate that the copolymers, when combined with nanostructured SeO_2 and BN nanocomposites, exhibit effective shielding capabilities against gamma rays.

According to the data presented in **Figure 4**, it can be observed that the highest rates of relative photon emission (RPE) were obtained at an energy level of 121.7817 keV. Furthermore, the maximum rate of RPE recorded in this study was

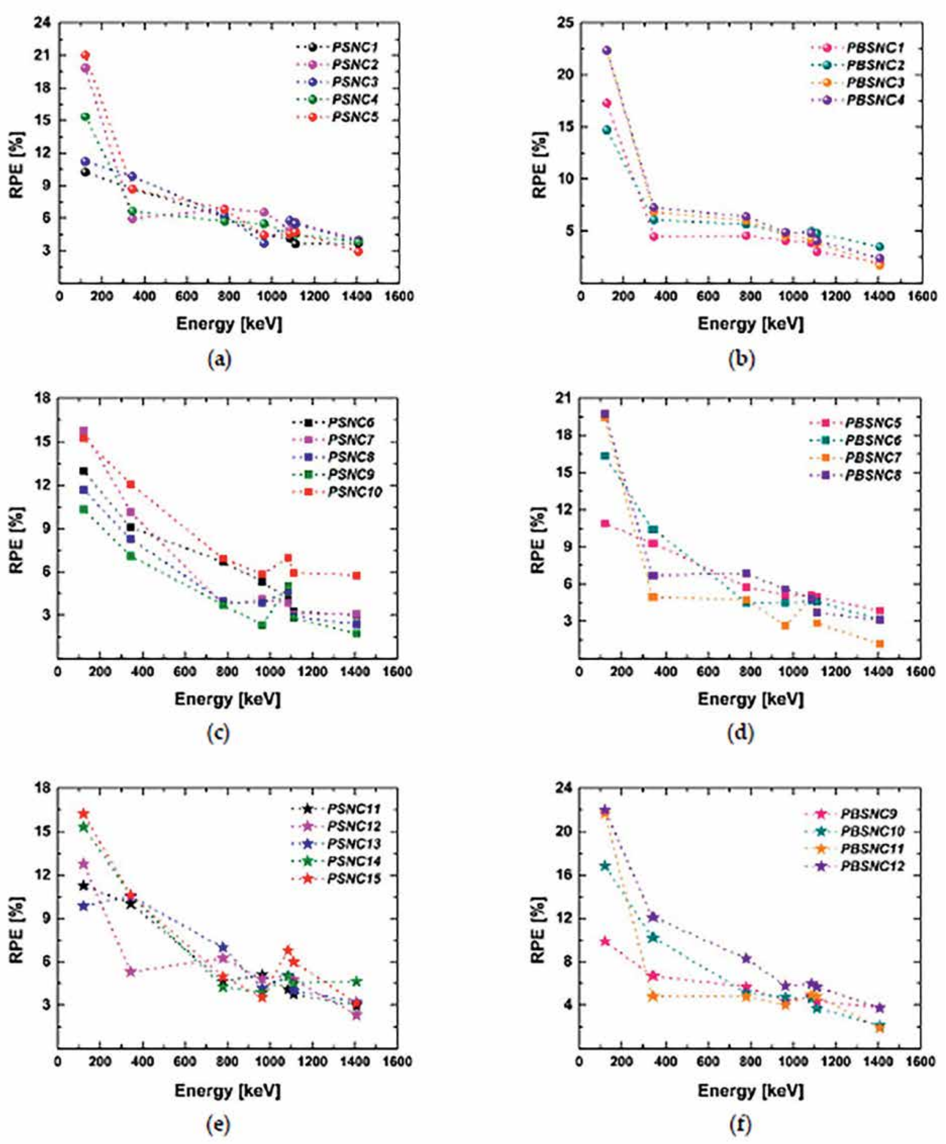

Figure 4.

The RPE rates of the PS-b-PEG copolymers blended with the nanostructured SeO_2 *and BN particles under a wide range of gamma irradiation energies.*

around 22.334%. Upon conducting an analysis of all PS-b-PEG copolymers blended with nanostructured SeO_2 and BN nanocomposites, it was determined that the ingredient with the largest abundance in the nanocomposites was responsible for exhibiting the highest rates of Radiation Protection Efficiency (RPE) and demonstrating optimal behavior in attenuating gamma radiation.

It is important to acknowledge that an increase in gamma radiation energy leads to a drop in RPE rates. This decrease in RPE rates provides confirmation that our findings align with other computed and experimentally confirmed attenuation parameters, including μ_L, μ_m, HVL, TVL, and MFP.

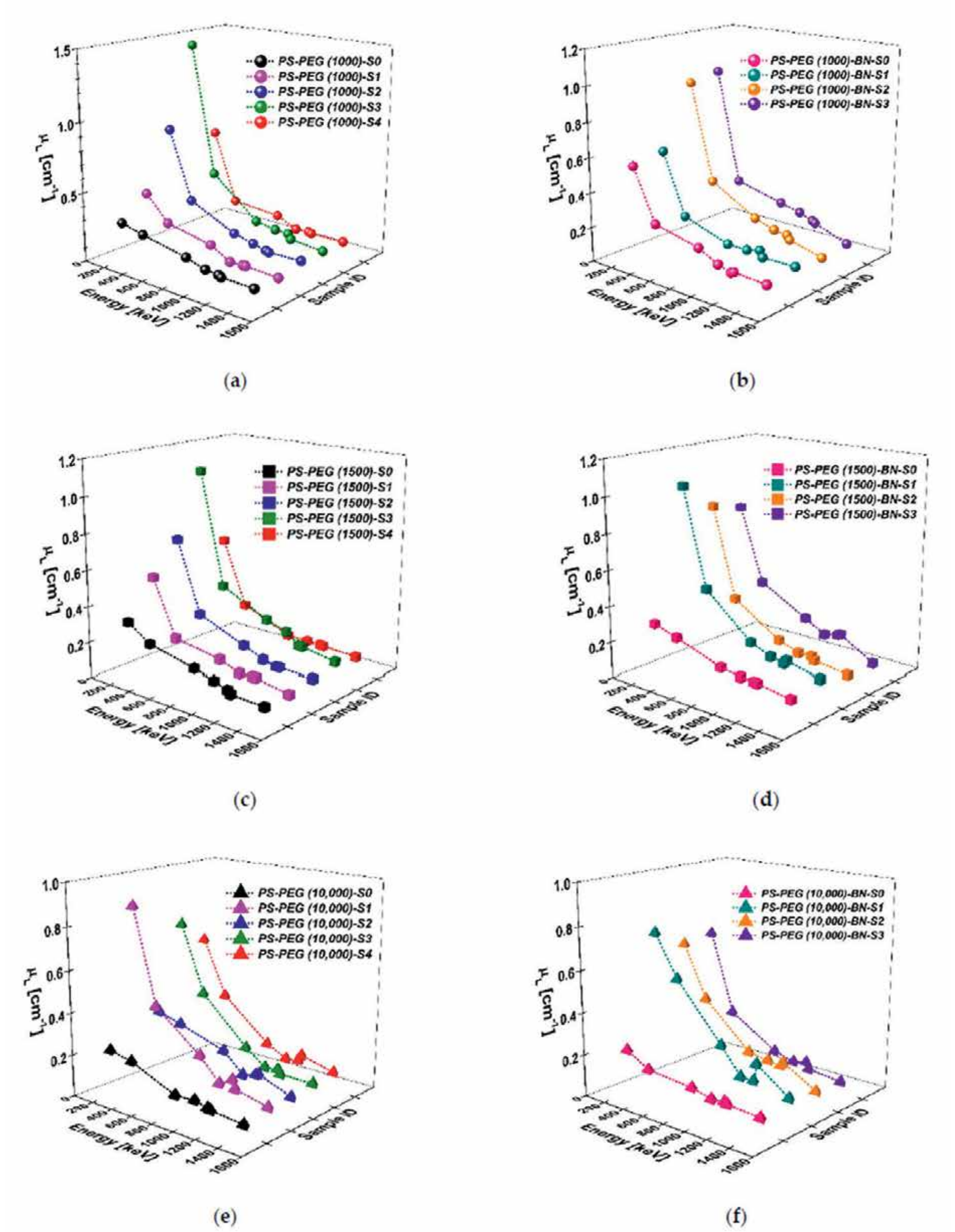

Figure 5.
The LACs values of the PbO doped the cross-linked PS-b-PEG block copolymers and the PbO doped the PS-b-PEG-BN nanocomposite materials at different gamma radiation energies (from 121.782 to 1408.006 keV of ^{153}Eu).

In their study, Cinan et al. [110] conducted research to investigate the effectiveness of gamma irradiation and the shielding properties on lead oxide (PbO) doped PEG (PS-b-PEG) block copolymers and PS-b-PEG-BN nanocomposite materials. In this study, the investigation focused on the gamma-ray shielding properties of cross-linked PS-b-PEG block copolymers and PS-b-PEG-BN nanocomposites, which were prepared by incorporating varying percentages of PbO. The copolymer was synthesized using emulsion polymerization techniques. The linear-mass attenuation coefficients (LACs-MACs), HVL, MFP, and RPE values of the samples were computed. Through the process of crosschecking the collected data from samples containing both PbO and BN, as well as samples without these substances, an observation was made. It was found that the addition of nano-powders of PbO and BN, in varying weight percentages, to the polymer mixture can serve as an effective shielding material for gamma rays. The incorporation of diverse forms of contributions, such as cement, polymer, and metal oxide, among others, plays a pivotal role in enhancing the hardness, durability, and radiation absorption capabilities of shielding materials. Polymer structures represent a significant category of materials employed in the study of radiation shielding due to their cost-effectiveness and low weight. In **Figure 5**, the LACs of PbO-doped cross-linked PS-b-PEG block copolymers and PbO-doped PS-b-PEG-BN nanocomposite materials are presented. The LACs were measured at various gamma radiation energies ranging from 121.782 to 1408.006 keV, using ^{153}Eu as the radiation source. Additionally, they serve as a basis for various research endeavors involving composites obtained by appending micro- or nano-oxides, among other substances, to explore the theoretical and experimental aspects of radiation attenuation.

5. Laser ablation of polyethylene and derivatives and applications

Laser ablation is one of the promising material processing approaches to the surface, internal modification, and nanomaterial generations of different composites from metal to transparent materials (see **Figure 6**). Over the past two decades, this technique is also well applied to various polymer-based materials for the fabrication of surface structuring from micron to nanoscale. Laser ablation of a polymer target starts with the absorption of incoming photons producing the heating and photoionization of the irradiated area. Subsequently, some polymer material can be torn away from the target as vapors, liquid drops, solid fragments, or as an expanding plasma

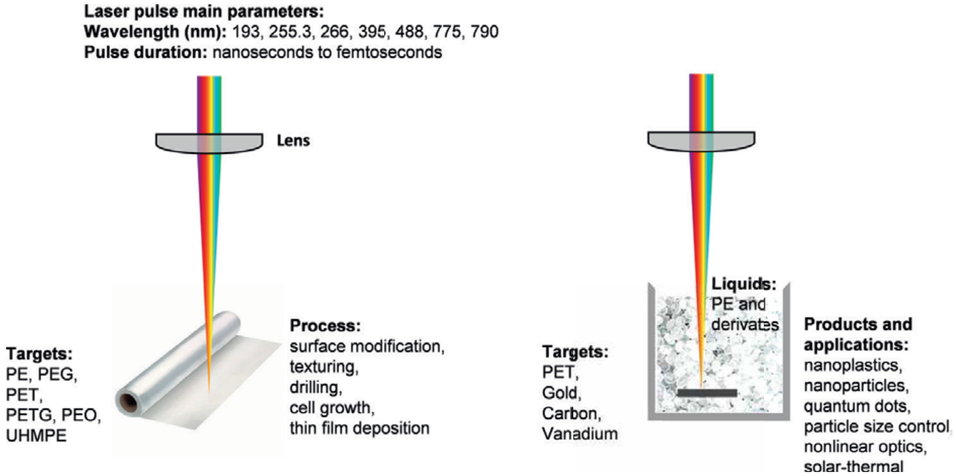

Figure 6.
Overview of laser ablation and applications of polyethylene and its derivatives.

plume resulting in the surface modification of polymers. Polymer materials are removed by laser ablation technique and could also be used for thin film deposition. In addition, laser ablation of materials in a polymer organic solution opens the door for nanomaterial fabrication with interesting properties. Many factors influence the materials processing performance such as laser parameters (wavelength, power, pulse duration, energy, peak power, etc.), material properties (compositions, absorption, thermal conductivity, thermal expansion coefficient, etc.), laser material interaction parameters (time, focal plane, number of the pulses, etc.), and laser interaction environment (compositions, viscosity, concentration, etc.). Different laser systems and influence factors of the laser interaction to PE and derivatives ablation have been studied in the literature.

The single pulse laser ablation process on PEG 1000 has been investigated by using a time-resolved fast photography technique [111]. The polymer target was ablated by an ArF excimer laser operating at 193 nm wavelength with a pulse duration of 20 ns and an applied fluence of 1.95 J/cm^2. The interaction area and the process were analyzed with a temporal resolution of 1 ns. They reported that the polymer material ejection from the interacted area is observed in two different time domains: development and removal of plasma (called primer ablation) in the range of 0–50 ns and secondary material ejection between approximately 1 and 100 μs. They assumed that the secondary material ejection is a result of thermal dissociation local melting and vaporization induced by the significant and very fast heating up of the polymer. The number of pulses and the pulse repetition effect on the etch performance of glycol-modified polyethylene terephthalate (PETG) were also investigated [112]. A UV laser operating at 255.3 nm with a pulse duration of 30 ns and a repetition rate from 0.75 to 15 kHz with fluences up to 0.59 J/cm^2 were tested. They observed that the mean etch rate (μm/pulse) increased slightly with the pulse repetition rate. It is also demonstrated that PETG removal rates (μm/sec) at 15 kHz were therefore at least 15 times faster than at 1 kHz due to the cumulative heating of the sample. The factors of focused spot size, pulse energy, and ablated hole diameter on laser ablation of polyethylene terephthalate (PET) were analyzed by using two different laser systems based on 266 nm nanosecond pulses and 775 nm femtosecond pulses [113]. The thickness of the PET samples played a key role in the estimation of the diameters of the holes machined. In addition, multiple pulse regimes showed different behavior. They demonstrated that smaller holes were achieved with multiple pulses and a lower fluence than with a single pulse with the same total energy. Another comparative study has been also reported in the literature. A Ti: Sapphire laser operating at 790 and 395 nm, and a mixture of both beams were used for the ablation of PE [114]. It has been observed that the etching time of the PE was shorter in the 395 nm case compared to the 790 nm laser. On the other hand, the mixture of the two colors resulted in even deeper an etching performance with the formation of isolated carbon and $C{-}O$ and $C{=}C{-}H$ bonds for all cases. The mechanisms of pulse laser ablation (400 ps pulse duration and 488 nm operation wavelength) of ultra-high-molecular-weight-polyethylene (UHMWPE) are investigated [115]. It is reported that the pulse laser irradiation induced a strong polymer dehydrogenation and molecular emission due to different C_xH_y groups having high kinetic energy and high charge state. In addition, it is also observed dehydrogenated material and carbon-like structures in the crater walls and at the bottom of the crater, respectively. In Ref. [116], a model was proposed to estimate the area and depth of laser ablation. An HDPE was studied by using a laser operating at 1064 nm wavelength with a pulse duration of 10 ns. Different laser powers and the number of pulses allowed to fabricate blind microholes. A model of quantitative area-depth approximation was proposed and confirmed based on gain factors determined by the experimental data with high accuracy.

Laser ablation of polymers is also useful for surface modification and thin film deposition for real-world applications. Different degrees of change to the wettability characteristics of UHMWPE was performed by using different laser operation wavelengths. The laser wavelengths at 1064 and 532 nm showed minimal change in the wettability characteristics but the laser wavelengths at 248 and 308 nm resulted in a marked improvement in the wettability characteristics of the UHMWPE. In particular, the UHMWPE irradiated by 308 nm, after 10 laser shots, reported a linear increase of the polymer wettability [117]. Third harmonics (355 nm) wavelength of solid-state Nd^{3+}:YAG laser used for pulse laser wet texturing of flexible PET. A successful texturing of the PET sample was achieved by using a laser fluence of 30 J/cm^2. The tungsten as a bottom electrode for the photovoltaic device was deposited onto the textured PET surface, and it was shown to enhance optical absorption properties [118]. A Kr–F excimer laser was also applied on the surface modification of the PET samples and investigated the surface tension, surface morphology, and surface chemistry properties of the laser-treated PET samples for biological evaluation. Laser-treated sample presented an enhanced cell behavior, especially cell proliferation and cell adhesion and modified cell morphology [119]. Laser-treated PET surfaces could be considered for biological applications, such as implantable biomaterial. Femtosecond laser pulses were also applied to create a sub-micrometer hole on a thin PET membrane that separated two electrodes. The cell placed onto this hole by negative pressure was punctured using subsequent femtosecond laser exposure to form a hole in the cell membrane. This application opened the way for electrophysiological measurements [120]. In the literature, smooth polyethylene oxide (PEO) thin films with a maximum thickness of 250 nm have been deposited by using a second harmonic (532 nm) Nd^{3+}:YAG laser with a pulse duration of 8 ns pulses and a laser fluence of less than 1.6 J/cm^2. A similar crystalline structure of the formation of PEO thin film compared to the target PEO pellet was confirmed. The molecular weight of the target was affected by the nature of the thin film because the target having a low molecular weight target created more particulate ejection compared with the high molecular weight targets [121].

Laser ablation of materials in liquid media is a simple, one-step, and one of the promising top-down nanomaterials synthesizing techniques. This approach is also well applied for nanoparticle fabrication from PE, as well as in the PE environment. Nanoplastics were fabricated from PET samples by using the laser ablation method to investigate the real environmental nanopollutants effects. The chemical/physical properties and stability of PET nanoparticles in different media were analyzed. The nanoplastics having 100 nm of average dimension presented heterogeneities in size, shape, and weak acid groups on their surface similar to photodegraded PET plastics. No toxic effects were observed *in vitro* studies in the short term. On the other hand, those nanoparticles were passed through an *in vitro* Transwell model of the intestinal epithelium [122]. PE solution as a liquid for pulse laser ablation of materials is also an attractive environment for novel nanoparticle generation. The synthetization of gold nanoparticles by using a second harmonic Nd^{3+}:YAG ns laser in PE glycol and chitosan for comparative study has been carried out in terms of particle size and optical properties. They indicated that the gold nanoparticles generated in PE were about a glycol environment of 7.49 nm in size, which was the smallest particle size compared to other solutions. The increase in the volume fraction of gold nanoparticles was also improved in PE glycol compared to other solutions. Nonlinear optical properties of the products, such as nonlinear refractive index and nonlinear absorption coefficient, were studied by using the Z-scan technique. The decrease in gold nanoparticles size generated in the PE glycol solution, as well as the increase in the volume fraction directly affected by the nonlinear coefficient compared to other solutions [123]. Nanosecond laser ablation of a graphite target in PEG 200 resulted from the synthetization of fluorescent carbon quantum dots. In this study, the high production efficiency of quantum dots was achieved by using two-step laser ablation (ablation of graphite target and then further ablation of suspension). 1064 nm laser pulse ablation was shown better effective compared to laser ablation with a 355 nm laser pulse. As a result, strong and broadband photoluminescence behavior of quantum dots was fabricated [124]. Vanadium nanoparticles were also synthesized by ablation of the Vanadium target using a 1064 nm laser with a pulse duration of 100 ns in PEG medium. High dispersion stability was presented for vanadium nanoparticles generated in a PEG medium acting as a stabilizing agent compared to vanadium nanoparticles generated in a water medium under similar conditions. The light-to-heat conversion of nanoparticles was then investigated and better performance from vanadium nanoparticles generated in PEG medium in terms of conversion efficiency of incident light-to-heat and thermal stability was obtained compared to nanoparticles generated in water [125]. This type of nanoparticle generated in a PEG medium is a potential candidate for solar-thermal application.

6. Conclusion

PE, a versatile polymer, finds extensive application across diverse industries and various aspects of our everyday existence. PE finds widespread application in various domains, including the following areas: PE is a commonly employed material in the manufacturing process of packaging materials, including but not limited to plastic bags, cling film, plastic bottles, plastic caps, and bags. In this chapter, we evaluated the use of PE and its derivatives as ion-exchange and ion-exchange membranes. We also presented information about the synthesis, exchange capacities, selectivity coefficient of ion-exchangers, and ion-exchange membranes containing cross-linked PE and PEG. The significant advancements in membrane technology and the growing interest in the past decades can be attributed to the extensive potential for practical application of ion-exchange membranes. Ion-exchange membranes are widely utilized in several industrial processes, including electrodialysis, electrodeionization, and electrochemical manufacturing, on a large scale. Simultaneously, ion-exchange membranes find application in electric power generation systems. The majority of commonly used and commercially available superabsorbent materials are derived from acrylic acid polymers and copolymers, which are derived from petroleum and are nonrenewable and nonbiodegradable. The broad applicability of SAPs can be attributed to several distinctive attributes. These include the relative abundance of opposing charges, the extent of counterion dissociation, and the presence of intramolecular and intermolecular hydrogen bonding, as well as the coulombic interactions that occur between opposing charges within the network structure. The beneficial characteristics of SAPs have rendered them highly sought-after in several domains, including agriculture and horticulture, selective adsorption, bio-separation, fertilizer coatings, controlled medication release, smart materials for biomedical devices, and cardiovascular disease treatment. The implementation of effective measures to mitigate the detrimental impact of ionizing radiation on human health and the environment is of utmost importance. The use of polymeric composites as neutron and gamma shields has increased significantly in recent years. Multiple researchers have proposed the construction of multipurpose shields by incorporating metal structures or synthesized minerals into these composites so that they can be utilized in photon fields. Boron-containing PE composites can be utilized as effective neutron shields due to their high neutron absorption cross section. The integration of many types of contributions, including cement, polymer, metal oxide, and others, significantly contributes to the improvement of the hardness, durability, and radiation absorption properties of shielding materials. Polymer structures are widely utilized in the field of radiation shielding because of their cost-effectiveness and lightweight nature, making them a prominent category of materials in this area of study. Laser ablation is effectively applied to a variety of polymer-based materials for micron- to nanometer-scale surface structuring. Ablation of a polymer target by laser begins with the absorption of incoming photons, which heats and ionizes the irradiated area. Some polymer material can then be torn away from the target as vapors, liquid droplets, solid fragments, or an expanding plasma plume, resulting in surface modification of polymers.

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Author details

Ahmet Hakan Yilmaz[1], Bülend Ortaç[2], Saliha Mutlu[2,3] and Sevil Savaskan Yilmaz[2,3]*

1 Faculty of Sciences, Department of Physics, Karadeniz Technical University, Trabzon, Turkey

2 UNAM-National Nanotechnology Research Center, Institute of Materials Science and Nanotechnology, Bilkent University, Ankara, Turkey

3 Faculty of Sciences, Department of Chemistry, Karadeniz Technical University, Trabzon, Turkey

*Address all correspondence to: sevily@ktu.edu.tr

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Chapter 6

Perspective Chapter: Morphological and Thermal Properties of Biodegradable Graft Copolymer LLDPE-g-MA/Gelatin Composites

Normurodov Nurbek Fayzullo Ugli
and Ashurov Nigmat Rustamovich

Abstract

In this work, a biodegradable graft copolymer based on linear low-density polyethylene grafted maleic anhydride and gelatin (LLDPE-g-MA/Gel) was formed by reactive mixing of functionalized polyethylene with gelatin to achieve finely dispersed blend morphology. Using a selection of components of the mixture, we studied its morphology and thermal properties. It was found that the thermal stability (initial temperature) of the composition decreases as the amount of gelatin increases due to the degradation of gelatin. In the temperature range of 400–500°C, the maximum rate of destruction of the graft copolymer increases significantly with increasing gelatin content. Samples with the same composition were selected using a Brabender plastograph, and a mechanical mixer, and when taken at different speeds, the morphological structure of the samples was determined to depend on their mixing speed. It was found that the morphological structure, with the increase in speed, the two components react well, and the particles become smaller.

Keywords: TGA, DSC, biodegradation, gelatin, glycerin, polyethylene, maleic anhydride

1. Introduction

The mixture of polyolefin with natural thermoplastics is of great interest not only from a scientific point of view but also because of the possibility of their practical application. Polymers are blended to produce polymer materials with new improved properties and to expand the range of polymer materials.

The unregulated advancement of morphology during the creation of biodegradable compositions using both synthetic and natural polymers hinders the potential for attaining satisfactory physical, mechanical, and operational traits. Several studies have reported the successful preparation of PE/gelatin blends using various techniques, such as solution casting, melt blending, and electrospinning. These studies have demonstrated that the addition of gelatin can improve the mechanical, thermal, and barrier properties of the blends [1–7]. Blending can improve a wide variety of polymer properties. However, while polymer blending is attractive for producing new materials, most polymer blends are incompatible. This is the reason for the difficulties of processing processes and the deterioration of the performance properties of such polymer mixtures [8, 9].

The packaging sector requires biodegradable materials. Despite the advantages such as production technology, flexibility in physical and mechanical properties, and cost-effectiveness, traditional synthetic polymers, especially polyolefin, present notable ecological challenges for the environment. To address this issue, researchers have investigated the use of biodegradable additives to improve the biodegradability of PE while maintaining its properties [10–15]. An examination of scientific literature suggests that addressing this issue involves employing biopolymers or combining them with synthetic polymers.

From this perspective, the enthusiasm for developing blends of gelatin with synthetic polymers remains strong [16, 17]. Simultaneously, it has been observed that the conventional mixing of gelatin with polyolefin in different ratios results in very low rates of biodegradability (up to 10%). Optimizing the blend's morphology by transitioning gelatin to a thermoplastic state slightly enhances this indicator.

Amidst the escalating environmental pollution crisis, significant research focus in academia and industry over the last two decades has been directed toward biodegradable polymers. These environmentally friendly polymers are acknowledged as promising substitutes for conventional polymers due to their ability to biodegrade, thereby mitigating waste pollution. Biopolymers, such as gelatin, chitosan, and starch, have attracted significant attention due to their biodegradability, biocompatibility, and renewability. Gelatin, in particular, is a protein-based biopolymer derived from animal collagen and has been extensively used in various applications, including food, pharmaceuticals, and cosmetics [18–21]. Biodegradable polymer-based blends/composites have been manufactured to enhance the various properties of original polymer components notably [22, 23]. The rate of crystallization and the level of crystallinity in crystalline polymers significantly influence their properties and ultimate applications. By altering the crystallization conditions of polymers capable of crystallizing, a wide range of morphologies and supramolecular structures can be achieved [24–29].

In this research work, LLDPE-g-MA/Gel copolymers based on maleic anhydride (MA) grafted polyethylene (PE), (LLDPE-g-MA) and gelatin (Gel) were obtained. Crystallinity structure, thermal stability, morphological structure, and hydrolytic degradation of neat PE, LLDPE-g-MA (5%), and LLDPE-g-MA/Gel composites and components were investigated and compared.

2. Materials and research methods

2.1 Materials

Research and experiments were carried out by the authors of the article in the laboratory of nanostructured polymer composite materials of the Institute of Polymer Chemistry and Physics within the framework of the scientific project aimed at the fundamental goal of “Nanocomposite polymer based on polyolefins - polymer blends materials produced in Uzbekistan” in 2022–2023.

Used in the work: linear low-density polyethylene (LLDPE) grade F-0320, d = 0.920 g/cm^3, MFI = 2.5 g/10 min (at a load of 2.16 kg). Producer—Shurtan Gas

Chemical Complex of the Republic of Uzbekistan; edible gelatin (GEL) grade P-200 (GOST 11293–2019). Producer JSC "MOGELIT," Belarus; maleic anhydride (MA) $C_4H_2O_3$, analytical grade, colorless rhombic crystals, M_r = 98.06 g/mol, was distilled at T_{bp} = 84.0°C/14 mm Hg, T_m = 60°C, ρ^{60} = 1.3140 g/cm^3.

2.2 Preparation of thermoplastic gelatin

To dissolve gelatin granules and make them thermoplastic, glycerin was added to distilled water and stirred until the same mixture was obtained; gelatin was added to the resulting mixture and mixed again, then heated in an oven at 80°C for 2 hours.

2.3 Functionalization

Functionalization of LLDPE with maleic anhydride (LLDPE-g-MA) was carried out on a Brabender plastograph (Plasticorder Brabender OHGDUISBURG Germany), with a cam speed of 98 rpm and at a temperature of 180 ± 5°C [30]. The concentration of maleic anhydride in the weld is 5.0% by weight.

Polymer blends based on LLDPE-g-MA and gelatin were obtained on a Brabender plastograph, for 20 min, at 50 rpm and 180 ± 5°C by adding plasticized gelatin to the LLDPE-g-MA melt.

2.4 DSC and TGA measurements

Thermal analyses of DSC and TGA of polymer blends were carried out in dynamic mode on a LINSEIS THERMAL ANALYSIS PT1600 [thermogravimetric analysis (TGA)] instrument (in air atmosphere) in the range from room temperature to 1000°C, guided by the requirements established by the ASTM E 1131 standard. The heating rate was 10°C/min, and the sample weight for analysis was from 2 to 100 mg.

2.5 Atomic force microscope (AFM)

The morphology of polymer mixtures was studied using atomic force (scanning probe microscope Agilent 5500) microscopy at room temperature. We used silicon cantilevers with a stiffness of 9.5 N/m and a frequency of 145 kHz. The maximum scanning area on AFM along X, Y is 15 × 15 μm^2, along Z it is 1 μm.

3. Results and discussion

3.1 TGA and DSC measurements

Thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) are commonly used techniques to investigate the thermal properties of materials. The results obtained from these measurements can provide valuable information on the thermal stability, decomposition behavior, and phase transitions of the materials under investigation.

The statement in the text suggests that the TGA measurements have revealed the presence of reaction products in the mixtures and initial components under investigation. The changes in the thermogravimetric characteristics, such as the initial degradation temperature (T_i) and the temperature of the maximum degradation rate (T_{max}), can indicate the formation of new compounds or the degradation of the original components.

Table 1 provides a summary of the T_i and T_{max} values for various compositions of mixtures and initial components. These values can be used to compare the thermal stability of different materials and to assess the effect of mixing on the thermal properties of the composite materials (**Figure 1**).

The TGA curves for the LLDPE-g-MA/gelatin blend reveal a thermal decomposition process occurring in three successive stages. The initial stage, within the temperature range of 100–200°C, involves the removal of absorbed and bound water molecules, resulting in a weight loss of less than 10%. The subsequent stage, occurring between 275 and 400°C with a weight loss ranging from 10 to 50 wt%, corresponds to the cleavage of peptide bonds within the gelatin macro chain [31]. The temperatures corresponding to the maximum rate of thermal decomposition of the gelatinous phase are within 250–280°C. A partial contribution to the weight loss of the composition in this temperature range is also made by the onset of LLDPE-g-MA dehydrogenation, the maximum rate of thermal decomposition (third stage) of which falls at a temperature of 450°C. The position of this peak practically does not depend on the content of gelatin in the composition; as the content of gelatin increases, it slightly shifts toward higher temperatures (up to 7°C). Temperatures, contingent on the anticipated rate of thermal decomposition of the gelatin phase, are identified within the range of 250–280°C. Within this interval, the initial stages involve LLDPE-g-MA dehydration, and an escalated rate of thermal decomposition (third stage) is observed at 450°C. The position of this peak remains essentially constant irrespective of the gelatin content; however, as the gelatin content rises, it notably increases at

No	Sample name and component ratio (wt%)	The temperature corresponding to the loss of mass is °C				Degree of crystallinity %
		Initial degradation temperature T_i, °C	Maximum thermal decomposition rates (T_{max}), °C			
			first stage	second stage	third stage	
1	LLDPE	186	100–200°C m(mg) ≤ 10%	275–400°C 10 ≤ m(mg) ≤ 50%	400–500°C m(mg) ≥ 50%	19.85
2	LLDPE-g-MA (5%)	242				18.15
3	LLDPE-g-MA/GEL 70/30	233				14.85
4	LLDPE-g-MA/GEL 60/40	223				13.32
5	LLDPE-g-MA/GEL 50/50	190				12.02
6	LLDPE-g-MA/GEL 40/60	201				10.4
7	GELATIN	97				—

Table 1.
Thermal characteristics of LLDPE-g-MA/gelatin blends.

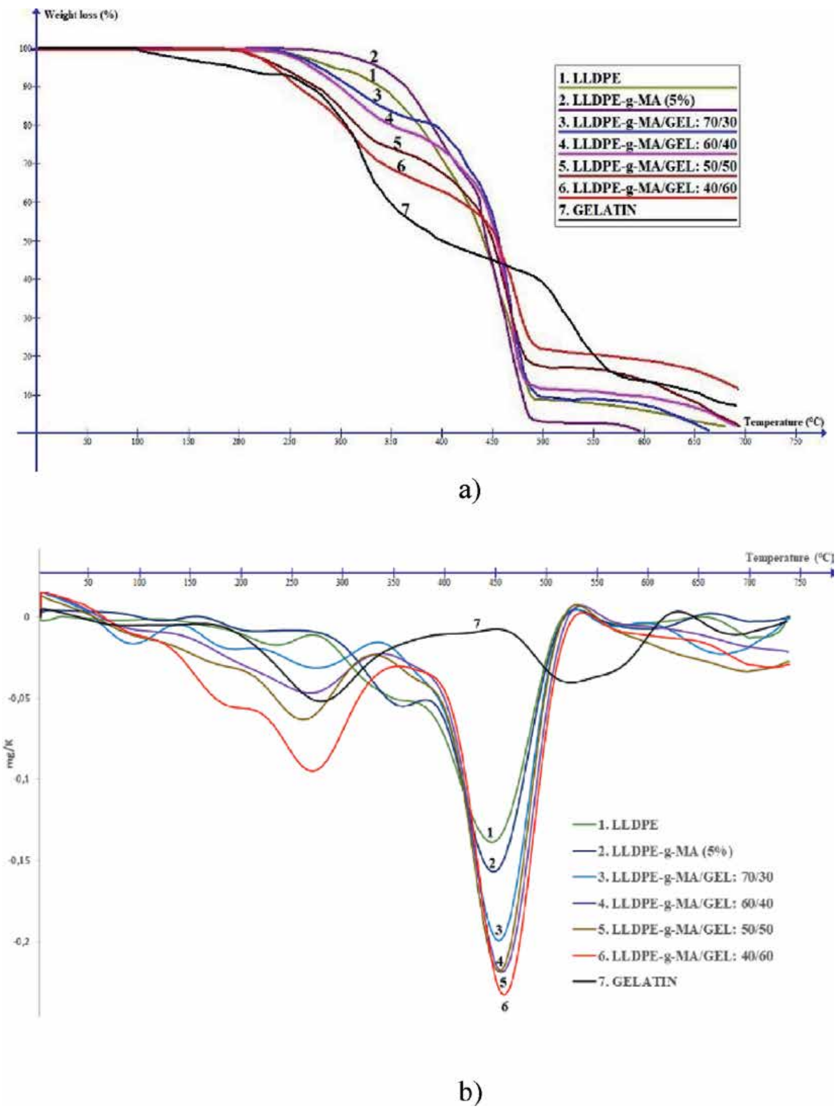
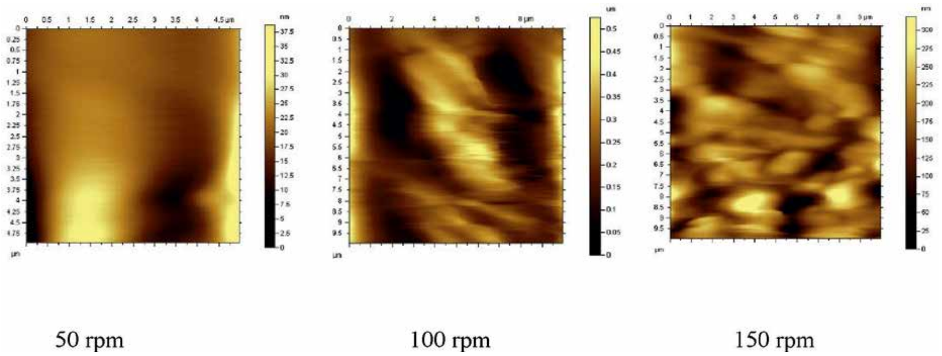


Figure 1.
Thermogravimetric curves (a) and the first derivative of the TGA curves (b) of the film sample.

higher temperatures (up to 7°C). To sum up the TGA data, it is evident that the formation of the graft copolymer LLDPE-g-MA/gelatin results in a reduction in thermal stability, with a Ti decrease ranging from 10 to 40°C depending on the gelatin content (30–60 wt%). Simultaneously, the maximum rate of thermal decomposition in the third stage substantially increases with higher biopolymer content (from −0.14 to −0.23 mg/K) due to the degradation of the grafted gelatin.

3.2 Atomic force microscope (AFM)

The use of atomic force microscopy (AFM) in the study of the morphology of the dispersed phase particles in polyethylene gelatin composites is a valuable technique



50 rpm 100 rpm 150 rpm

Figure 2.
Atomic stress microscope images of samples taken at 50 rpm, 100 rpm, and 150 rpm.

as it allows for high-resolution imaging of the surface topography and can provide information on the size, shape, and distribution of the particles.

The results presented in **Figure 2** suggest that the mixing speed has a significant effect on the size and shape of the dispersed phase particles. Specifically, increasing the mixing speed from 50 to 150 rpm leads to a reduction in particle size and a change in particle shape. This suggests that the use of higher mixing speeds may be beneficial in achieving a more uniform distribution of the dispersed phase particles in the composite material, which can ultimately lead to improved physical and mechanical properties.

Overall, the use of AFM in conjunction with varying mixing speeds provides valuable insights into the morphology of composite materials and can aid in the development of improved processing methods for these materials.

In order to prevent the coalescence of particles of the dispersed phase of gelatin in polyethylene and to ensure uniform morphological distribution, the samples were mixed at three different speeds and studied under atomic force microscopy (AFM). Coordinating speeds varied from 50, 100, and 150 rpm (**Figure 2**). The phase analysis of the size and distribution of the dispersed phase particles shows that the dispersed phase particles become smaller in size and have different shapes when the speed is increased.

AFM studies show that in the structure of the drawn samples, the dispersed phase particles of the second component increased in velocity, and the distribution improved with less coalescence. Thus, microscopic studies have shown that the obtained polymer mixtures have a structural morphology that helps to improve the physical and mechanical properties of the obtained materials.

4. Conclusion

Grafting thermoplastic gelatin onto maleated polyethylene facilitates the creation of uniform compositions with a more refined dispersion of the gelatin phase within the polyethylene matrix. The thermal decomposition of the LLDPE-g-MA/gelatin blend manifests in three stages linked to the presence of water molecules, degradation of the gelatin, and degradation of the polyethylene phases.

The introduction of gelatin induces a significant reduction in the onset temperature of decomposition, shifting it from 242–201°C, while the temperature range for the degradation of the polyethylene matrix remains relatively unchanged. Notably, the maximum degradation rate of the graft copolymer within the 400–500°C range sees a noticeable increase, moving from −0.14 to −0.23 mg/K with higher gelatin content. Microscopic examinations reveal a structural morphology in the obtained polymer mixtures that contributes to enhancing the physical-mechanical properties of the resulting materials.

Overall, the findings of this study demonstrate the potential of using reactive mixing to achieve finely dispersed blend morphology in biodegradable copolymers. The observations regarding the effect of gelatin content and mixing speed on the thermal and morphological properties of the copolymer provide valuable insights into the development of improved processing methods for these materials.

Author details

Normurodov Nurbek Fayzullo Ugli* and Ashurov Nigmat Rustamovich
Uzbekistan Academy of Sciences, Institute of Polymer Chemistry and Physics,
Tashkent, Uzbekistan

*Address all correspondence to: nnf7nnf7@gmail.com

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Chapter 7

Regulatory and Safety Requirement for Use of Recycled Polyethylene in Food and Cosmetic Packaging

Ramez Labib, Ripal Amin and Lisa Hoffman

Abstract

Post-consumer resin, or PCR, is the technical term for recycled plastic. Plastic waste is collected, sorted, and re-purposed to make various types of new packaging. This includes ocean plastics, which are post-consumer resins made from plastics that are headed to oceans but picked up for recycling before they end up in oceans. Polyethylene terephthalate (PET) and high-density polyethylene (HDPE) are the most frequently recycled polymers where PET and HDPE bottles comprise 97.1% of the United States plastic bottle and packaging market. The greatest challenge to using PCR plastic is the potential chemical migration of contaminants from the plastic packaging into the package contents. This potential chemical contamination is often higher in PCR material as compared to virgin material due to the migration from and to the plastic during its lifetime. There are regulatory requirements to ensure safety of PCR in food packaging, but there are no clear guidelines for cosmetic industry to ensure their safe in cosmetic packaging. In this book chapter we will summarize the challenges for using recycled polyethylene, the global regulatory requirements for their use in food packaging and learning that can be applied for their safe use in cosmetic packaging.

Keywords: cosmetic packaging, food packaging, post-consumer recycled plastic, quantitative risk assessment, threshold of toxicological concern, dermal sensitization threshold

1. Introduction

Plastic use is omnipresent in our society, where it's used in durable consumer goods as well it's use for packaging in cosmetics, food, and self-care products. The discarding of these plastics tends to be separated into three streams: solid waste for landfilling/disposal, recycling, and incineration use. Growing concern over the potential environmental impact of disposed plastic waste has led to the widespread use of post-consumer recycled (PCR) plastic. PCR is a type of recycled plastic material that is obtained by collecting, sorting, cleaning, and processing plastic waste that has been discarded by consumers after use [1]. This waste can come from a variety of sources, including plastic bottles, containers, bags, and other types of packaging. The collected plastic waste is then sorted based on its type, color, and quality. It is then cleaned and processed into small pellets or flakes that can be used to create new plastic products. The resulting PCR material can be used to make a wide range of plastic products, including new containers, bags, packaging materials, and other items for a variety of industries [2].

The use of PCR plastic is important for both sustainability and the circular economy. By using PCR plastic, companies can reduce their reliance on virgin plastic production, which conserves natural resources, reduces greenhouse gas emissions, and promotes resource efficiency [3]. In addition, the use of PCR plastic promotes a circular economy by creating a closed-loop system where materials are reused and repurposed, rather than discarded after a single use [1, 3]. The quantity and characteristics of the plastic used in PCR is variable and often depends on geographical location, but the most recycled plastics include polyethylene terephthalate (PET), high-density polyethylene (HDPE), low-density polyethylene (LDPE), polypropylene (PP), and polystyrene (PS) [1]. These types of plastics are often found in products such as water bottles, grocery bags, food containers, and packaging materials [1, 4]. Of the four main types of polymers, PET and high-density PE are the most frequently recycled polymers, where HDPE and PET bottles compromise 97.1% of the United States plastic bottle and packaging market and both plastics are used in 98.6% of the recycled bottles [5].

While the use of PCR materials represents an important step towards more sustainable plastic manufacturing, there are several challenges associated with its use including maintaining the quality and consistency of the recycled material [1]. PCR plastics can vary in quality and consistency depending on the source material, the recycling process, and other factors. This can make it difficult to use PCR plastics in certain applications, such as high-performance products, where consistent material properties are critical [6]. The availability and supply of PCR plastics can also be a challenge. PCR plastics are typically more expensive to produce than virgin plastics, and there may be limited supplies of certain types of PCR plastics, depending on factors such as collection and recycling infrastructure, consumer behavior, and market demand [1, 6].

However, the greatest challenge to using PCR plastic is the potential chemical migration from the plastic packaging into the package contents [4]. This potential chemical contamination is often higher in PCR material as compared to virgin material due to the migration from and to the plastic during its lifetime [1, 4, 7]. To address this challenge, proper sorting, cleaning, and processing of PCR plastics is critical. However, absence of chemical decontamination included in a strict recycling process will result in post-consumer use plastic which may be contaminated with chemicals [4]. Contaminants can originate from the original products made from the plastic, such as food packaging, containers, or household items as well as external sources, such as additives used during the manufacturing process [1]. Potential residual chemicals that may be found in PCR materials includes traces of substances such as dyes, pigments, and food residue that leached into the recycled plastic during use and persisted in the material after recycling [7] as well as plastic additives such as flame retardants, plasticizers, UV stabilizers, antioxidants, and heavy metals [8]. The presence and concentration of these contaminants can vary depending on the specific recycling process, the type of plastic being recycled, and the level of quality control and sorting implemented during recycling operations [7]. Many of these contaminants have the potential to pose a risk to human health and therefore should be evaluated from a safety perspective prior to using PCR materials.

The use of recycled plastics for food contact is subject to various regulations. For example, in EU, the European Food Safety Authority (EFSA) assess the use of food contact materials containing recycled plastic and only those approved by the European Commission are permitted as outlined in various regulations including EC 1935/2004 [9], EU 10/2011 [10]. Other industries such as cosmetics are increasingly using PCR plastics to reduce the environmental footprint of their products [4]. However, there is no direction on the safety evaluation of PCR materials for use in cosmetic packaging. Therefore, in this chapter we will summarize the EU and US regulatory requirements for use of rPET in food packaging and learnings that can be applied to the safe use of rPET for cosmetics.

2. Classification of input steam and recycling process

The input materials for the recycling processes play an important role in the risk assessment of the recyclates and has an impact on the contamination levels [11]. The different approaches to the recycling of plastic packaging materials can be categorized into three distinct processes:

Primary recycling (input materials are referred to as class 1) is the recycling of industrial scrap produced during the manufacture of food contact plastic where the material has a low likelihood for contamination and provided that the material has been evaluated already by scientific groups, e.g., EFSA or FDA, it can be reused in cosmetic and food packaging materials like virgin materials [12]. Secondary recycling (input materials are referred to as class 2 or 3) are waste discarded by a given consumer or industry, after its use, is recovered and processed to manufacture new products, which do not necessarily have the same purpose as the original product [12]. The input materials under secondary recycling are divided up into two classes:

- Class 2: are post-consumer materials for well-known applications [11], which are recollected as pure grade by the recycler and the contamination levels are typically low and the variation in the contamination levels is small (e.g., milk bottles or pure collected shampoo bottles).

- Class 3: are post-consumer materials that typically have a wide range of contaminants at high levels as those are recollected from mixed plastics collections with unknown history of use with potential cross contamination from consumer misuse [11]. As a result, having good sorting efficiency is critical to establish homogenous fractions with a low potential for contamination [11].

Tertiary recycling where input materials are referred to as Class 4, use thermochemical methods to process the materials, organic or non-organic, which will be molecularly disintegrated and transformed into new products. The resulting materials are normally used as primary materials (oligomers and monomers), fuels or are co-processed with other materials. In the case of plastics, tertiary recycling allows the production of naphtha or monomers, which are further processed to produce new plastics [12].

In the case of class 2 and class 3 materials, contaminants might be present, and they require further control prior to using in recycled plastic. In general class 1 and class 4 materials are assumed to be safe and can be used with virgin polymers to remanufacture new packaging for food and cosmetic products.

The recycling process of polyethylene comprises the collection, sorting, cleaning, reprocessing by melting and then producing new products from the recycled polyethylene. It is critical for recycled plastics used in food or cosmetics to undergo a decontamination process in the recycling of plastics. Various contaminants can be present in recycled plastic including those introduced from consumer misuse (i.e., petrol or domestic-use pesticides) or unintentionally added during collection and recycling processes (e.g., detergents from the washing step), or newly formed during the recycling process (e.g., degradation products of plastic components). A so-called challenge test can be performed to assess the efficiency of the decontamination step during recycling where plastics are "spiked" (i.e., chemicals are added) with a set of representative surrogate contaminants at known concentrations [13]. Then, the intentionally contaminated plastics undergo a recycling process and analyzed afterwards to illustrate that the surrogate contaminants have been reduced to levels that do not pose any health concerns.

Polyethylenes are common as food packaging components. In Europe, post-consumer recycling of PET into new PET bottles has been well established over past two decades and as a result, the procedures put forward for recycling of polyolefins are largely based on those developed for PET [14]. However, PET and polyolefin polymers have different material properties, which also includes the diffusion behavior of substances in the polymer where polyethylene has poorer functional barrier properties and a faster migration of absorbed contaminants within the polymeric material. This results in polyethylene being potentially highly contaminated due to fast sorption of contaminants, with higher molecular weight than those typical for PET [15]. As a result, polyethylene needs to be decontaminated to a high degree, since the migration of residual contaminants into food is likely to be fast and hence of higher concern [15]. Also, polyethylene has a reduced thermal stability which results in higher amounts of degradation products formed during the decontamination processes, especially the one based on thermal desorption, which is used most widely used [15]. Moreover, polyethylene has an application in a wide range of products, including fragranced cleaning agents as they often contain several different additives. Consequently, it becomes prone to contamination with a variety of substances to a more significant degree when compared to PET, which makes processing of polyolefins more challenging. This has major effects for proper decontamination procedures for polyolefins recycling, which should differ from those used for PET.

3. Potential contaminants in recycled polyethylene

Some data has been published on contamination levels of post-consumer recyclates. In one study, bottle samples from household waste were collected from five different sites where twenty-one rHDPE pellets samples of the bottle fraction were investigated towards post-consumer substances [16]. A total of seventy-four substances were identified at concentrations above 0.5 mg/kg which are not detected in virgin HDPE. Most of the substances are identified as constituents from consumer goods and cosmetic products including saturated fatty acids, phthalate esters, hydrocarbons, preservatives, monoterpenes and sesquiterpenes. The highest concentrations ranging from 50 to 200 mg/kg were found for limonene, diethylhexyl phthalate and the isopropyl esters of myristic and palmitic acid. In a follow-up study, seven rHDPE and eight rPP samples were analyzed [17] where limonene was detected as main substance in concentrations up to 100 mg/kg as well as phthalates esters, alkanes, 2,6-di-*tert*-butyl-4-hydroxytoluene and oligomers, but there was no indication of bottle misuse (e.g., for the storage of toxic chemicals).

Another study evaluated the contamination levels of rHDPE flakes from milk bottles recollected in the UK [18]. Unsaturated oligomers were the predominant contaminants in washed rHDPE flakes which were also found in virgin HDPE pellet samples. Like virgin HDPE, the concentrations of both decene and dodecene were around 20 mg/kg. Concentrations of the saturated oligomers were detected in slightly higher concentrations versus virgin HDPE. Both limonene and antioxidant additives di-*tert*-butylphenol were detected in the post-consumer rHDPE flakes. Unknown substances were found in one sample at 130 mg/kg and 40 mg/kg, respectively, which is an indication of consumer misuse.

A more recent analysis was conducted by Danish Environmental Protection Agency where they analyzed contaminants found in HDPE and PP [19]. The primary sources of waste incorporated samples from household polymer-sorted plastic, materials derived from fishing equipment, and collected plastic waste originating from oceans, rivers, and beaches within the Indian Ocean region. The utilization of GC/MS screening led to the identification of a considerable quantity of non-aromatic aliphatic compounds, with the majority being found in the saturated state. The non-aromatic, saturated aliphatic compounds may potentially stem from the degradation of polymer chains. This category of components is prevalent and stands as the most significant group detected. The presence of these components within the samples is semi-quantitatively estimated to range between 0.05% and 0.7%, respectively. One sample also contained several aromatic compounds of aliphatic functionality (MOAH, mineral oil aliphatic hydrocarbons), such as 5-phenyltridecane. These compounds do not originate from the degradation of the polymer chain, which does not have an aromatic functionality, but rather may be from components that migrated into the plastic throughout its lifetime while being used by the consumer. Also, different phthalates (DBP, DIBP, BBP and DEHP) were detected in several samples at relatively low levels (in all cases below 0.005%). The ICP/MS screening also revealed a small number of metals that have migrated into the product simulant including 0.5 mg/kg aluminum.

4. Food regulatory requirements for PCR

Food contact regulatory requirements for recycled plastics around the world are complex with regions like US and EU establishing the stringent requirements for authorization of recycled plastic, whereas regions like Australia, China, India do not have formalized regulations. However, it is essential to mitigate the risks associated with migration of chemical substances from recycled plastics into food, which could pose a health risk. The primary hindrance to the utilization and growth of recycled plastics in food contact applications stemmed from the absence of a regulatory framework that would permit and ensure the safe use of post-consumer materials for food purposes.

4.1 United States Food and Drug Administration (US FDA)

The US Food and Drug Administration (FDA) regulates the food contact materials including food packaging as per the Food, Drug, and Cosmetic (FD&C) Act. FD&C Act defines a food contact substance (FCS) as any substance that is intended for use as a component of materials used in manufacturing, packing, packaging, transporting, or holding food if such use of the substance is not intended to have any technical effect in such food [20].

FDA has adopted the Food Contact Notification (FCN) program as part of the Food and Drug Administration Modernization Act of 1997, where a manufacturer or supplier of a food contact material may, in most cases, submit a notification to FDA identifying the food contact substance and its intended conditions of use, and providing information to support the conclusion that the substance is safe for the intended use. If the FDA does not raise any objections within 120 days of receiving a notification, the manufacturer or supplier can proceed to market the product [20].

US FDA requires the prior approval of recycled plastic for food contact application via FCN before marketing by the manufacturer. FDA requires the manufacturer to submit the following information on post-consumer recycled resin. If all criteria were met, the FDA would issue a "Letter of No Objection" (LNO).

1. A comprehensive account of the entire recycling procedure, encompassing details on the origin of the PCR plastic as well as a description of any implemented processes used to guarantee that only plastic which originally adhered to relevant regulations is recycled. Additionally, an overview of the measures taken to prevent any contamination of recyclable plastic at any stage, whether prior to collection for recycling or during the actual recycling process [21].

2. The outcomes of any conducted tests aimed at illustrating the efficacy of the recycling procedure in eliminating potential incidental contaminants. When utilizing recycled material as a replacement for plastic produced from virgin sources, it's critical to demonstrate that there is no possibility of contamination with substances other than food. This can be achieved through rigorous source control on the input material, or to showcase the efficacy of the recycling process in effectively removing contaminants via surrogate testing. If necessary, further migration testing or migration modeling may be carried out to demonstrate that the recycling process effectively eliminates potential incidental contaminants to a level that does not allow each contaminant to exceed 0.5 ppb, a threshold that the FDA deems as negligible exposure when employing recycled plastics for food packaging [21].

3. An explanation outlining the anticipated utilization scenarios for the plastic material. This entails providing details such as the intended usage temperature, the specific type of food that will have contact with the plastic, the duration of this contact, and whether the plastic designed for food contact is intended for single-use or repeated application [21].

4.2 European Union

The European Union (EU) has established comprehensive obligations and legislations that apply to a wide range of food contact materials and substances. All food contact materials fall within the scope of two European legislations: Regulation (EC) 1935/2004 (framework regulation) and Regulation (EC) 2023/2006 on good manufacturing practices. The framework regulation mandates the adoption of specific measures and guidelines for the groups of food contact materials including virgin plastics (Regulation (EC) 10/2011) and recycled plastics (Regulation (EC) No. 2022/1616) [22].

4.2.1 Regulation (EC) No. 1935/2004

This regulation emphasizes that food contact materials must not pose any health risks or cause alterations in the smell, composition, color, or taste of the food.

Furthermore, this regulation requires that food contact materials are manufactured in compliance with good manufacturing practice (GMP) as regulated by Regulation (EC) No. 2023/2006. It also specifies labelling and traceability (one step forward and one step back) requirements for food contact materials [9].

4.2.2 Regulation (EC) No. 2023/2006

Regulation 2023/2006 lays down the rules on good manufacturing practice (GMP) that apply to all group's food contact materials. This regulation is applicable at all stages of the production and distribution process of food contact materials excluding the production of raw materials. This regulation outlines the requirements for quality assurance, quality control systems and documentation practices [23].

4.2.3 Regulation (EC) No. 10/2011

This regulation, also referred to as plastic implementation measures (PIM) is the most comprehensive legislation in the field of food contact materials. This regulation establishes specific requirements to ensure the safety and suitability of plastic materials used in food contact applications. This regulation applies to plastic materials and articles, whether composed exclusively of plastics (plastic monolayers or plastic multilayers) or plastic parts combined with other materials (multi-material multi-layer) [10].

The regulation covers various aspects such as permissible substances, migration limits, and testing methods to assess the compliance of plastic materials with food safety standards. It aims to safeguard consumer health by minimizing the migration of harmful substances from plastic packaging or utensils into food. Adherence to these regulations is essential for manufacturers, importers, and distributors involved in the production and distribution of food contact plastic materials within the EU market [10].

4.2.4 Regulation (EC) No. 2022/1616

This regulation governs the specific measures for recycled plastic materials and articles intended to come into contact with food. This regulation applies to post-consumer plastic waste, which refers to plastic waste generated by consumers rather than being a byproduct of another product's production [24]. Recycled plastic must originate from an authorized recycling process that follows a Quality Assurance System (QAS) as defined in Regulation (EC) No. 2023/2006 [23]. The QAS is responsible for ensuring that the recycled plastic meets the specific requirements outlined in the authorization [25].

Furthermore, a recycling process must effectively monitor and evaluate both the initial plastic input and the resulting recycled plastic, adhering to predetermined criteria that guarantees compliance with the safety requirements outlined in the Framework Regulation for authorization i.e., plastic input complies with the Regulation (EC) No. 10/2011 for plastic materials and the GMP Regulation (EC) No. 2023/2006. The main principles mirror those captured in the US FDA criteria and include [25]:

- Comprehensive detailing of the procedure.
- Thorough characterization of the initial material.

- Evaluation of the process's decontamination effectiveness using a surrogate challenge test after washing.

- In depth characterization of the recycled plastic.

- Explanation of the intended application.

- Demonstration of regulatory conformity.

- Identification of key process steps.

- An explanation of quality assurance mechanisms.

It aims to promote the circular economy by encouraging the safe and sustainable use of recycled plastics in food packaging applications.

4.3 Canada/Mexico

Canada has established guidelines for utilizing recycled plastics in food contact application and used the similar criteria outlined by the US FDA. Instead of implementing an official regulation, Canada opts for a case-by-case evaluation of technologies and issues a Letter of No Objection (LNO), similar to the United States [26].

Mexico does not have official regulations for recycled plastic. They have reviewed the concept and have adopted the US FDA guidance on use of recycled plastic in food packaging. In 2016, the National Standardization and Certification Organismo instituted a voluntary standard, NMX-E-263-CNCP, which addresses post-consumer recycled PET for food packaging. This aligns with guidance provided by the US FDA [26].

4.4 South America

In 2002, the Mercosur countries (Argentina, Brazil, Paraguay, Uruguay, and Venezuela) launched a collaborative initiative to comprehensively evaluate available data regarding recycling technologies for utilizing post-consumer plastics in food contact applications. In 2007, the Mercosur countries implemented legislation concerning post-consumer PET materials through resolution GMC 30/07 in 2007. This resolution incorporates a combination of guidelines from both the US FDA and EFSA [26].

The Mercosur resolution incorporates the following essential components:

1. Processed postconsumer PET material must fulfill all regulatory requisites applicable for Virgin PET for food contact.

2. Authorized processes must undergo registration under the respective national governments.

3. Allocation of responsibilities includes:

    - Proposed recycling technologies must successfully complete surrogate challenge testing for validation.

- Conducting tests to evaluate usage conditions.

- Plants must be approved by the applicable sanitary authority.

- Mandating package processors to be registered entities, maintain comprehensive documentation, and possess complete capabilities for package traceability.

- The food processor is only permitted to utilize authorized material/packages and must be capable of demonstrating complete traceability.

4.5 Central America

Costa Rica: In 2017 Costa Rica implemented decree 40393/2017, which mandated the adaptation of the Costa Rican Standard RTCR 480-2016 for incorporating post-consumer PET in food contact. This standard drew its foundation from the guidelines set forth by the US FDA [26].

4.6 Asia-Pacific

4.6.1 Australia and New Zealand

Australia and New Zealand shares the food standards-setting procedures. The regulatory framework for food contact materials in Australia and New Zealand is comparatively less specific when compared to other jurisdictions like the European Union and the USA. In both countries, the emphasis is placed on the overarching legal obligation to ensure food safety. Currently, the Australian Packaging Covenant have indicated that the food standards code is currently under revision and more details will be included on use of recycled plastic [26].

4.6.2 China

Food contact materials (FCMs) are regulated by the general food safety law (FSL) adopted in 2009. The Chinese food packaging standards are primarily guided by the recently developed GB 4806.1-2016 (referred to as the "framework" standard) along with GMP standard GB 31603-2015. China does not have formalized recycled packaging regulations however, the Chinese government has directed the Chinese National Health and Family Planning Commission to initiate a re-evaluation of the current packaging regulations and other regulations pertaining to food additives [26, 27].

4.6.3 Japan

Japan's National Institute of Health Sciences conducted a thorough review of scientific literature and the US FDA/EU guidelines, leading to the drafting of a proposed guidance document. Subsequently, on April 27, 2012, the Ministry of Health, Labor, and Welfare authorized the guidance document 0427 NO 2 [26].

4.6.4 India

Regulations regarding plastic, PET and recycled PET usage in India are issued by the Bureau of Indian Standards. The document IS 14534:1998 titled "Guidelines for Recycling of Plastics" indicates the step-by-step procedures for the recovery and recycling of plastic waste. It explains in detail the procedures to follow for the selection, segregation and processing of waste/scrap that are suitable for recycling [28].

5. Cosmetic use of PCR

There is a high uncertainty in the cosmetics market, both among manufacturers and recycling companies, about the use of PCR in cosmetics packaging. According to Regulation (EC) No. 1223/2009 on cosmetic products, manufacturers should only put on the market safe products. However, there are no guidelines on safety assessment of recycled material and conditions for which they may be used.

The major challenge for the recycling of plastic waste into new cosmetic packaging applications is that post-consumer substances or degradation products from the polymer or from polymer additives substances could migrate into the cosmetic product and result in adverse effects upon consumer use. The use of post-consumer recyclates in cosmetic packaging should not raise safety concerns for the consumer. Therefore, use of recycled plastic in cosmetic packaging requires assessment of potential contaminants and ensuring that levels would not pose a risk to consumer's health [11].

Two recent studies have evaluated the use of recycled polyolefins (including HDPE) in cosmetic applications [11, 19]. The first step in the assessment was to identify substances found in the rHDPE and perform a quantitative risk assessment. The type of substances identified includes chemicals from the polymer, which were also found in the reference packaging made from virgin HDPE or chemicals from previous fillings or from cross-contamination during recollection or recycling. There were also unidentified or unknown chemicals found in rHDPE.

An exposure-based risk assessment was then performed to determine whether use of the rHDPE in cosmetic packaging would result in potentially any adverse systemic toxicity. For the identified substances a specific toxicological evaluation was done on each of the substances and determining the dose that is likely to be without any appreciable risk of deleterious effect during a lifetime and compared to the consumer exposure from daily use of cosmetic product (a rinse-off or a leave-on application). For the non-identified substances, a specific toxicological evaluation is not possible, and evaluation should be done using the principle of Threshold of Toxicological Concern Concept (TTC) [29]. The TTC is a pragmatic risk approach which is based establishing a threshold for human exposure below which there is an extremely low probability of any appreciable risk to human health from any chemical. A worst-case assumption was used that the unknown substance should be evaluated as a potentially genotoxic substance with a the relevant TTC value is 0.0025 μg/kg bw per day [29].

In both studies an exposure scenario was then carried out for rinse off products (e.g., shampoo) as well as leave on applications (e.g., body lotion). For rinse off products, it was assumed that 99% of the applied product is washed off and only 1% remaining has potential to be absorbed and become systemically available. For leave on products, one study used an adult body lotion [11] while other did an assessment for a baby lotion [19] where in both cases, it assumed that the cosmetic product remains on the skin and the resorption into the body will be assumed as 100%. Based on both assessments, it was demonstrated that rHDPE may be reusable as packaging for rinse off cosmetic products, but using genotoxicity TTC for unidentified substances, the rHDPE was not supported for use in leave on body lotions (including adult and baby).

In a more recent study [4] a safety assessment was conducted on rHDPE that was approved for food packaging to evaluate its use in cosmetic packaging using the guidelines published by EFSA on the use of recycled plastics. The highest concentration (Cmod) of representative chemical contaminants, that would not exceed the genotoxic TTC, when migrating from rHDPE packaging into the cosmetic formulation was predicted using a mathematical modeling software (MIGRATEST®EXP). A comparison was then made between the Cmod values of representative chemical contaminants to the EFSA-reported residual concentration (Cres) of each contaminant in the rHDPE. It was demonstrated that for each of the cosmetic product/packaging combinations evaluated, the modeled values were clearly lower for Cmod than Cres. This illustrates that the recycling process could effectively reduce potential contaminants of rHDPE to levels that would not pose systemic toxicity to consumers. In addition to systemic toxicity, the authors also investigated skin sensitization, where they assumed a worst-case scenario that 100% of each representative chemical contaminant migrates into the cosmetic formulation from rHDPE. The consumer exposure level for each contaminant was then calculated based on the dose per unit area. This was then compared with a value of 64 $\mu g/cm^2$ which is the acknowledged dermal sensitization threshold (DST) for reactive materials. In each case, it was demonstrated that there was no appreciable risk of skin sensitization as the migration of each representative chemical contaminant from rHDPE into each cosmetic formulation was significantly less than the DST. It was concluded that the rHDPE can be safely used in all cosmetic products (leave-on and rinse-off applications). This exposure-based approach can be used to assess additional polyethylene plastics to determine if they can be safely used for personal care products.

6. Conclusion

In recent years sustainability has become a key trend, with awareness on recycling everyday products and minimizing energy consumption becoming the norm in the average consumer's household. The use of post-consumer recycled (PCR) plastic is important for both sustainability and the circular economy. By using PCR plastic, companies can reduce their reliance on virgin plastic production, which conserves natural resources, reduces greenhouse gas emissions, and promotes resource efficiency. The greatest challenge to using polyethylene plastic is the potential chemical migration of contaminants from the plastic packaging into the package contents. In the food industry there are regulatory requirements to ensure safety of use of recycled polyethylene in the intended food application. In addition to food, the cosmetic industry has a very heavy impact on packaging [30]. Cosmetic companies are setting ambitious sustainability goals which includes use of recycled PCR plastic [31] adding the need for regulatory requirements or industry guidance to ensure the safety of use of PCR in cosmetic products.

Author details

Ramez Labib*, Ripal Amin and Lisa Hoffman
Avon Products International, Global Product Safety, Suffern, NY, USA

*Address all correspondence to: ramez.labib@avon.com

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