Mehaničko recikliranje jednokratnih maski za lice

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DIPLOMSKI STUDIJ KEMIJSKE TEHNOLOGIJE

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UNIVERSITY OF SPLIT

FACULTY OF CHEMISTRY AND TECHNOLOGY

GRADUATE UNIVERSITY STUDY OF CHEMICAL TECHNOLOGY

MATERIALS

MECHANICAL RECYCLING OF SINGLE-USE FACE MASKS

DIPLOMA THESIS

Petra Brajković

Parent number: 311

Split, October 2022.

TEMELJNA DOKUMENTACIJSKA KARTICA

DIPLOMSKI RAD

Sveučilište u Splitu

Kemijsko-tehnološki fakultet u Splitu

Diplomski studij kemijske tehnologije, smjer Materijali

Znanstveno područje: Tehničke znanosti

Znanstveno polje: Kemijsko inženjerstvo

Tema rad je prihvaćena na 25. sjednici Fakultetskog vijeća Kemijsko-tehnološkog fakulteta, održanoj 18. ožujka 2022.

Mentor: doc. dr. sc. Miće Jakić

MEHANIČKO RECIKLIRANJE JEDNOKRATNIH MASKI

Petra Brajković, 311

SAŽETAK: U ovom radu ispitan je utjecaj mehaničkog recikliranja na svojstva jednokratnih maski za lice. Jednokratne maske za lice su mehanički reciklirane koristeći jednopužni ekstruder. FTIR, TG i DSC korišteni su kako bi se odredila struktura i toplinska svojstva svakog sloja jednokratne maske za lice, jednokratne maske za lice prije i nakon recikliranja. FTIR analiza je potvrdila da je polipropilen glavni polimer sadržan u svakom sloju jednokratne maske za lice. Utjecaj recikliranja jednokratnih maski za lice na toplinska svojstva istražen je diferencijalnom pretražnom kalorimetrijom i termogravimetrijskom analizom. Neizotermnom termogravimetrijom utvrđeno je da recikliranje jednokratnih maski za lice nema utjecaj na toplinsku stabilnost. Primjenom diferencijalne pretražne kalorimetrije utvrđeno je da recikliranje utječe na kristalnost jednokratnih maski za lice.

Ključne riječi: jednokratna maska, mehaničko recikliranje, ekstrudiranje, polipropilen

Rad sadrži: 64 stranice, 48 slika, 9 tablica, 38 literaturih referenci

Jezik izvornika: engleski

Sastav Povjerenstva za obranu:

1. Prof. dr. sc. Ladislav Vrsalović - predsjednik

2. Izv. prof. dr. sc. Sanja Perinović Jozić - član

3. Doc. dr. sc. Miće Jakić – član - mentor

Datum obrane: 28. listopada 2022.

Rad je u tiskanom i elektroničkom (pdf format) obliku pohranjen u Knjižnici Kemijsko-tehnološkog fakulteta u Splitu, Ruđera Boškovića 35.

BASIC DOCUMENTATION CARD

DIPLOMA THESIS

University of Split

Faculty of Chemistry and Technology in Split

Graduate study of Chemical Technology, Orientation: Materials

Scientific area: Technical Sciences

Scientific field: Chemical Engineering

Thesis subject was approved by Faculty Council of Faculty of Chemistry and Technology, session no. 25 held on March 18th 2022

Mentor: Ph. D. Miće Jakić, Assistant Professor

MECHANICAL RECYCLING OF SINGLE-USE FACE MASKS

Petra Brajković, 311

ABSTRACT: In this paper, influence of mechanical recycling on the properties of a single-use face masks was examined. Single-use face masks were mechanically recycled by using a laboratory single-screw extruder. FTIR, TG and DSC were used to determine the structure and thermal properties of each layer of the single-use face mask, single-use face mask before and after recycling. FTIR analysis confirmed that polypropylene is the main polymer that composes each layer of the single-use face mask. The influence of recycling of a single-use face mask on its thermal properties was investigated by differential scanning calorimetry and non-isothermal thermogravimetric analysis. It was found by non-isothermal thermogravimetry that recycling does not affect thermal stability of single-use face masks. By using differential scanning calorimetry, it was determined that recycling affects the crystallinity of single-use face masks.

Keywords: single-use face mask, mechanical recycling, extrusion, polypropylene

Thesis contains: 64 pages, 48 figures, 9 tables, 38 references

Original in: English

Defence committee:

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Defence date: October 28th 2022.

Printed and electronic (pdf format) version of thesis is deposed in Library of Faculty of Chemistry and Technology in Split, Ruđera Boškovića 35.

Master thesis was prepared at the Department of Organic Technology, at the Faculty of Chemistry and Technology in Split, under the supervision of doc. dr. sc. Miće Jakić, in the period from May to September 2022.

''Noting in life is to be feared, it is only to be understood. Now is the time to understand more, so that we may fear less.''

Maria Skłodowska-Curie

By virtue of my family, boyfriend Zoran and friends I have achieved a lot in life, including this paper. Thank you for every tear, laugh, motivation, word of support and every moment of joy that you have given me.

I had the privilege of having the best colleagues at the Faculty of Chemistry and Technology in Split, the ones that backed me up in every situation. This is just the beginning of a beautiful friendship that began 2017 when I enrolled KTF.

I would also like to thank my mentor doc. dr. sc. Miće Jakić for his patience, help and selfless effort during this period.

Although we may find it difficult to admit, the professors are the ones who have built our way of thinking and solving challenges and I am grateful to them for that.

Let the song Nobody is crazy to sleep by Crvena Jabuka live on in the hearts of the yellow sharks from KTF, who will always be fans No. 1 in our hearts and stadiums.

THE ASSIGNMENT OF WORK:

1. Identification of the single-use face masks main material by using Fourier-transform infrared spectroscopy (FTIR), Differential Scanning Calorimetry (DSC) and Thermogravimetric Analysis (TG).

2. Mechanically recycle single-use face masks by laboratory single-screw extruder and characterize the samples after extrusion process.

3. Conclude can the single-use face masks be mechanically recycled and evaluate how extrusion process affects the properties of the final material.

SAŽETAK

U ovom radu ispitan je utjecaj mehaničkog recikliranja na svojstva jednokratnih maski za lice. Jednokratne maske za lice su mehanički reciklirane koristeći jednopužni ekstruder. FTIR, TG i DSC korišteni su kako bi se odredila struktura i toplinska svojstva svakog sloja jednokratne maske za lice, jednokratne maske za lice prije i nakon recikliranja. FTIR analiza je potvrdila da je polipropilen glavni polimer sadržan u svakom sloju jednokratne maske za lice. Utjecaj recikliranja jednokratnih maski za lice na toplinska svojstva istražen je diferencijalnom pretražnom kalorimetrijom i termogravimetrijskom analizom. Neizotermnom termogravimetrijom utvrđeno je da recikliranje jednokratnih maski za lice nema utjecaj na toplinsku stabilnost. Primjenom diferencijalne pretražne kalorimetrije utvrđeno je da recikliranje utječe na kristalnost jednokratnih maski za lice.

Ključne riječi: jednokratne maske za lice, mehaničko recikliranje, polipropilen

SUMMARY

In this paper, influence of mechanical recycling on the properties of a single-use face masks was examined. Single-use face masks were mechanically recycled by using a laboratory single-screw extruder. FTIR, TG and DSC were used to determine the structure and thermal properties of each layer of the single-use face mask, single-use face mask before and after recycling. FTIR analysis confirmed that polypropylene is the main polymer that composes each layer of the single-use face mask. The influence of recycling of a single-use face mask on its thermal properties was investigated by differential scanning calorimetry and non-isothermal thermogravimetric analysis. It was found by non-isothermal thermogravimetry that recycling does not affect thermal stability of single-use face masks. By using differential scanning calorimetry, it was determined that recycling affects the crystallinity of single-use face masks.

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INTRODUCTION

Lack of environmental awareness and education causes plastic pollution and recycling of face masks leads to better sustainable waste management. Appropriate recycling methods for face mask waste need to be carried out, otherwise it will create a long-term environmental risk with a great impact on ecosystems and human health. Most often, face masks have a direct impact on the environment cause after use they end up on landfill, in oceans, rivers, public places and in the environment. Moreover, the decomposition of face masks creates microplastic contaminants that can be released in watercourse.[1]

Separation of components that a mask is made of is difficult, and as a result masks end up being incinerated, disposed in landfills which leads to environmental pollution and ecosystem disruption.[2]

Face masks could be disinfected and sterilized for direct reuse, but in that case filtering effectiveness is reduced, better solution is recycling.

Today, various methods of recycling face masks have been developed and there are various applications of the material after recycling. Material that can be used in concrete, as a separator for aqueous rechargeable batteries, material for the production of furniture etc.

In this paper, mechanical recycling of single-use face masks was performed by a laboratory single-screw extruder. After extrusion, samples were analysed by using Fourier-transform infrared spectroscopy, differential scanning calorimetry and thermogravimetric analysis.

1. GENERAL PART

1.1. Face mask

In early modern Europe covering the nose and mouth had been part of traditional sanitary practices against contagious diseases and primarily about neutralizing miasma in the air through perfumes and spices held under a mask. By the 18^{th} century, those practices become marginal.[3]

British surgeon Joseph Lister postulated in 1867 that wound disease was caused by the germs of microscopically small living entities. In 1880 surgeons devised the strategy of asepsis that aimed to stop germs from entering wounds because hands, instruments and operator's exhalations were also risky. Carl Flügge bacteriologist discovered that respiratory droplets carried culturable bacteria. Johan Mikulicz, head of the surgery department from the University of Breslau (Poland) 1897 started to wear "a piece of gauze tied by two strings to the cap, and sweeping across the face to cover the nose and mouth and beard" and in the same time surgeon from Paris, Paul Berger also began to wear a mask in the operating room. The face mask was a strategy for total wound sterility. Face masks became widespread, in the period 1863-1969 over 2/3 of surgeons in US and European hospitals wore masks and by 1935 most of them wore masks. During Manchurin plague (1910.-1911.) and the influenza pandemic (1918.-1919.) face masks became mandatory means of protection.

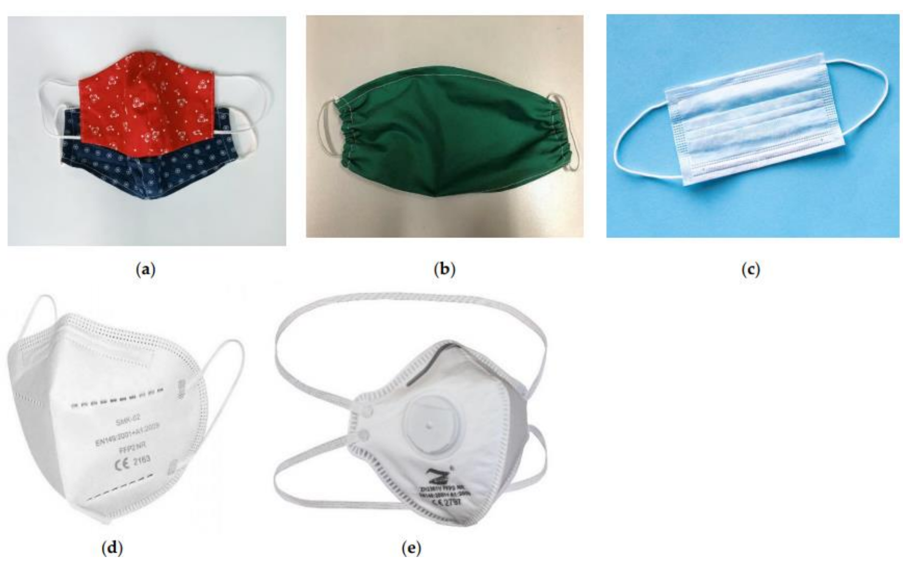

Figure 1. a) cloth mask, b) fabric mask, c) single-use face mask, d) FFP2 mask and FFP3 mask[4]

In later years, the rationale for wearing masks moved beyond their original use in the operating theatre and they continue with developing in the field of medicine. Face masks were made of several layers of cotton gauze held by a metal frame and most masks were washable. Researchers tested the filtering efficiency of reusable masks with experiments involving the culture and found that some masks offer protection from infection if they are used properly. In the 1960s synthetic materials were used in the production of face masks and these filtering masks could be used once because their synthetic fabric would deteriorate during sterilization. However, some studies were sponsored by the industry and they found a new synthetic mask. In 1975 studies showed that reusable mask made from four-ply cotton muslin is a better option in comparison with disposable paper masks and synthetic respirators. Further investigation showed that washing reusable masks can increase their bacterial filtering efficiency by tightening their fibres. The substitution of reusable masks with disposable face masks was a part of aggressive marketing campaigns.[3]

Mask Type	Standards	Filtration Effectiveness	
Single-Use Face Mask 	**China**: YY/T0969	3.0 Microns: ≥95% 0.1 Microns: X	
Surgical Mask 	**China**: YY 0469	3.0 Microns: 95% 0.1 Microns: 30%	
	USA: ASTM F2100	**Level 1**	**Level 2**
		3.0 Microns: ≥95% 0.1 Microns: ≥95%	3.0 Microns: ≥98% 0.1 Microns: ≥98%
	Europe: EN 14683	**Type I**	**Type II**
		3.0 Microns: ≥95% 0.1 Microns: X	3.0 Microns: ≥98% 0.1 Microns: X
Respirator Mask 	**USA**: NIOSH (42 CFR 84) **China**: GB2626	**N95/KN95**	**N99/KN99**
		0.3 Microns: ≥95%	0.3 Microns: ≥99%
	Europe: EN 149:2001	**FFP1**	**FFP2**
		0.3 Microns: ≥80%	0.3 Microns: ≥94%

Figure 2. Standards, ratings and filtration effectiveness for different types of face masks[4]

Fig 1 shows various types of face masks. Standards, ratings and filtration effectiveness for single-use face mask, surgical mask and respirator mask are presented in *Fig 2*.[4]

The consumption and demand of face masks has increased worldwide due to COVID-19 pandemic, because every day 6600 million masks are used.

Mayor requirements for face mask:

- Block input and output bacteria and virus
- Must provide > 95% efficacy
- Cheap, recyclable and sustainable
- Fit and comfortable to face
- Avoid leakage and allow normal breathing [4]

Table 1. Material used in production of face mask and their products and properties[4]

Polymers	Products	Properties
PP	Nonwoven melt blown and spunbond fibers	Low cost, lightest weight among all synthetic fabrics due to its low density and specific gravity, ability to filter dry particulates, high chemical (alkali and acid) resistance, ease of processing, recyclability, modifiable inherent hydrophobicity, good mechanical strength, abrasion resistance, and micropore distribution uniformity make PP a promising option for manufacturing face masks.
PE	Meltblown nonwoven fibers	PE with different densities, including high-density PE, low-density PE, and linear low-density PE, can be made. Good chemical resistance, lightweight, and hydrophobic. PE is easier to extrude than PP due to high shear sensitivity and higher melting temperature of PP.
PET	Spunbond nonwoven fibers	High tensile modulus, strength, and heat stability, but less cost-effective than PP and more difficult to recycle.
PA 6	Spunbond nonwoven fibers	Fiber lightness and high melting temperature, but unsuitable for face masks due to water absorption.
CA	Electrospun nanofibrous membranes	High filtration efficacy, low thickness, hydrophobic, low production cost, biodegradable, high water stability, but soluble in organic solvents.
PVA	Nanofibrous membranes	Lightweight, biodegradable, cost-effective, washable and reusable.
PLA	Nanofibrous membranes	Biodegradable, cost-effective, favorable mechanical properties and filtration efficiency of 99.99%.
PTFE	Air filter membranes	Lightweight, hydrophobic, great chemical stability, high surface fracture toughness, and high heat resistance. Because of its strong C-C and C-F bonds, PTFE membrane is extensively utilized as an air filter membrane with high filtration.
PAN	Waterproof membranes	High cost, significant variations in fiber diameters and mat morphologies, chemical and thermal stabilizations.

Synthetic thermoplastic polymers with uniform nanopore structure, good bonding ability and low cost are the main materials in the production of face masks. Although polypropylene and polyethylene are the commonly used materials in the production of face masks, other polymers shown in *Table 1* can be used as well. Nylon has a high affinity for particulates and adequate air permeability but it may create thermal discomfort. That is the reason why nylon is combined with nanoporous PE which has a filtration efficiency of 99.6% at high temperatures and sufficient cooling capabilities. The filtration efficiency of polyester and nylon is in the range of 5-25% and for PP is 6-10% but it can be increased by triboelectrically charging. PVA electrospun layer is replaced with a polypropylene inner layer due to massive hydroxyl bonds in PVA. Also, PP has disadvantages such as CO_2 emission during its production and the inability to withstand high heat. Water resistance is one of the requirements of face masks and PTFE is used in the production of waterproof membranes. Due to its high cost and membrane porosity new alternative, polyvinylidene fluoride electrospun membrane and hydrogel electrospun mat is used to create a waterproof membrane. Also, graphene oxide/gelatin and PAN have hydrophobicity and high filtration efficiency. For hydrophobic membranes PET, PP, PE and polyvinylidene chloride are used.[4]

Single-use face masks (medical masks) are classified according to three levels of protection: level one (low-barrier face mask), level two (moderate-barrier face mask) and level three (high-barrier face mask) where each level has different specifications. The certification of the European Standards Organisation (EN) is mandatory for a mask to be qualified as a surgical mask. Medical masks must meet specifications for filtering according to EN as bacterial filtration efficiency (BFE), submicron particulate filtration efficiency (PFE), etc. For level one requirements for BFE and PFE are $\geq$ 95%, while for levels two and three requirements are $\geq$ 98%.[4]

Disposable face masks are produced by using non-woven polypropylene fabric. More precisely, spun-bond polypropylene and melt-blown polypropylene are used as raw materials for surgical and non-surgical face masks.[5]

1.1.1. Polypropylene

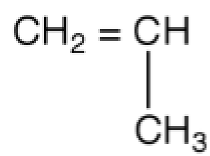

Figure 3. Propylene monomer[6]

Polypropylene (PP) is a polymer derived from the olefin monomer propylene through additional polymerization. The structure of propylene monomer is shown in *Fig 3* and the macromolecule of PP contains 10 000 to 20 000 monomer units.[6] In the process of polymerization, a catalyst, an initiator, heat or high-energy radiation are added to polymerize propylene molecules into long polymer molecules or chains. Isotactic (crystallizable) PP is the most broadly used form with catalysts that produce crystallizable polymer chains with good mechanical, thermal and physical properties. Atactic (non-crystallizable) PP is a by-product of semi-crystalline PP production with poor thermal and mechanical properties commonly used in adhesives as sealants.[7, 8]

Fig 4, *5* and *6* show polypropylene molecule in isotactic, syndiotactic and atactic forms. Since polypropylene has the lowest density among commodity plastics it is used in a wide variety of applications. Furthermore, it has a high melting point and relatively good resistance to impact. As a result of altering the chain tacticity and distribution, the average chain lengths, the incorporation of a comonomer into the polymer chains and the incorporation of an impact modifier into the resin formulation, properties of PP can be varied.[6, 7]

An isotactic PP has all the methyl groups attached to every second carbon atom in the chain in the same side of the winding spiral chain molecule. On the other hand, in syndiotactic PP pendant methylene groups are attached to the polymer backbone chain in an alternating manner. In atactic PP pendant groups are located randomly on the polymer backbone. Only isotactic PP is used as a useful plastic material.[6] The differences in stereoregularity influence the physical properties of the polymers, accordingly atactic PP has melting point at about 75 °C and is amorphous, while isotactic PP has melting point at about 160 °C and is crystalline.[9]

PP is characterized by excellent mechanical, thermal and physical properties when it is used in room–temperature applications. Calcium carbonate, talc, glass fiber and mica are the most widely added fillers and reinforcements to polypropylene resin to attain cost-effective composite mechanical properties. Fillers such as calcium carbonate and talc are used as extenders to produce a less-costly material but also can ameliorate impact and stiffness. [6, 7]

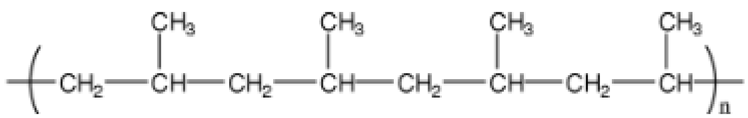

Figure 4. Isotactic PP[6]

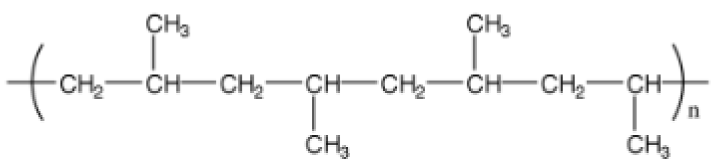

Figure 5. Syndiotactic PP[6]

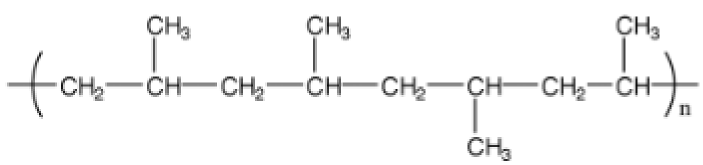

Figure 6. Atactic PP[6]

Polypropylene as nonwoven fibre can be used in medicine for the production of sanitary products, sterilization wraps, meshes and surgical growns.[10] Also, polypropylene fibre is used in water filters and membranes for the production of fresh water from sea water.[11] Polypropylene fibres can improve the properties of concrete such as good resistance to rutting, less reflection cracking and prolonged fatigue life by providing three-dimensional reinforcement of concrete. Higher fibre content does not mean better characteristics of fibre,

fibre should be added to the asphalt mix in a ratio of about 2.7 kg/t. Additionally, optimal fibre content is connected with the analysis of the mixture composition and properties of the polypropylene fibres. Concrete reinforced with polypropylene fibre leads to sustainable development and is used in the production of fountains, door surrounds, skateparks, ferry ports and sculptures.[12, 13]

The global polypropylene market is segmented by applications into: extrusion coating, blow moulding, other extrusion, film, fibre, injection moulding and others.[14]

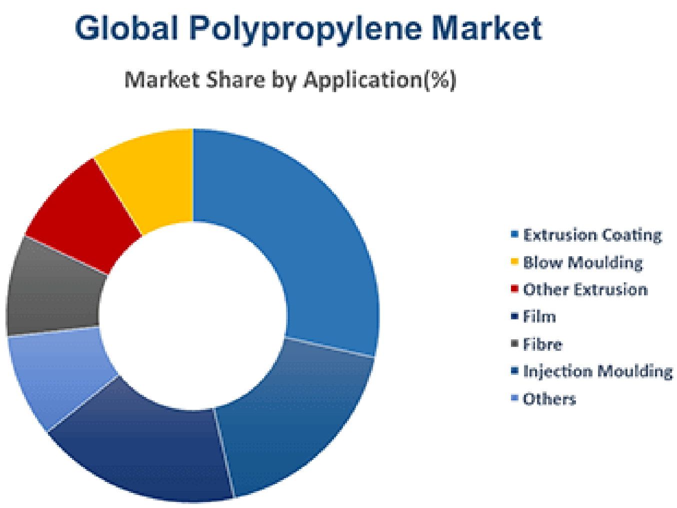

Figure 7. Global polypropylene market [14]

As can be seen from the chart (*Fig 7*) injection moulding is the leading application sector for polypropylene (nearly 40% of the total global application). In injection moulding polypropylene is softened, conveys with a screw and pushed through a runner system into a cavity or multiple cavities of a mould. In the end mould is cooled. Polypropylene moulded plastic parts are used in the food industry.[14, 15]

1.2. Environmental problems connected with single-use face mask

The increasing use of face masks is adding an enormous amount of plastics into the fresh and marine water, increasing the landfill which consequently causes environmental pollution. Also, negatively affect human and animal health.[5, 16] Therefore, the new environmental challenge is to reduce the environmental impact by reducing the use of masks.[16] Face masks often end up in rivers, seas or oceans harming marine and wildlife as can be seen in *Fig 8*. The adsorption of organic and inorganic nutrients on plastic waste in water can provide a supportive environment for bacteria.[5, 17] They can also be seen on streets due to mismanagement and unawareness.[5]

Figure 8. Problems with face masks[16]

About 1.56 billion face masks entered the oceans in 2020. Plastic pollution represents a major global challenge in the 21st century.[18]

One single-use face mask releases around 1.5 million microplastic particles by weathering actions.[5] Polypropylene face masks are a new source of microplastic <500 µm in length due to photodegradation, corrosion and immersion in water causing contamination of the ecosystem.[19, 20] Aging due to mechanical action and natural exposure promotes the release of microplastics and it is known that the middle layer of the face mask release more microplastics in comparison with the inner and outer layer. In addition to microplastic face masks also release heavy metals such as Pb, Cu, Mn, Fe, Ti, Ca, Sb, Zn and organic pollutants in environmental media.[19, 21] Also, microplastic from face masks can influence methanogenic communities in aerobic digesters. Future research should be focused on the investigation of the kinetics of microplastics released from disposable face masks into the aqueous environment. Therefore, the implementation of a new waste management system limiting the number of masks entering the environment should reduce pollution.[20] Consequently, with the increase in production and consumption, microplastics released from polypropylene face masks will continue to increase in the next period.[18]

Face masks in a municipal solid waste can interfere with high solids anaerobic digestion in waste management facilities. Laboratory experiments were conducted with an organic fraction of municipal solid waste supplemented with disposable face masks and the results showed that their presence could have a negative effect on methane kinetics and productivity.[20] Toxic gases, dioxin and furan are generated during the incineration of waste masks.[5] Lack of environmental awareness, poor waste management systems and education can cause plastic pollution. Only proper disposal, redesigning and recycling of face masks lead to sustainable waste management.[20] On the other hand, often recycling face masks require high-cost and time-consuming pre-treatments such as the separation of plastic materials and disinfection.[22]

1.3. Study of recycling potential of single-use face masks during Covid-19

Recycling polypropylene masks leads to a better approach in terms of the circular economy.[16, 22] Lack of environmental awareness, poor waste management systems and education can cause plastic pollution. Only proper disposal, redesigning and recycling of face masks lead to sustainable waste management.[21] On the other hand, often recycling face masks require high-cost and time-consuming pre-treatments such as the separation of plastic materials and disinfection.[22]

Figure 9. Life cycle of single-use face masks[23]

As can be seen in *Fig 9* life cycle of face masks have a big impact on the environment, most often after use face masks end up in landfill, oceans or rivers, public places, trash bins and in the environment. To limit the impact on the environment due to defined problems, face masks have to be recycled.

Face masks could be disinfected and sterilized for direct reuse, but in that case filtering effectiveness is reduced. Hence, it is not recommended. A better solution is to recycle materials from which masks are made.[17]

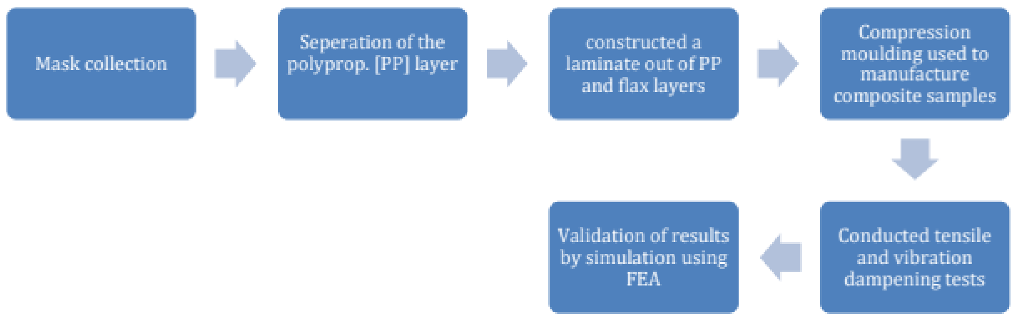

Figure 10. Methodology of producing FlaxPP material[17]

Fig 10 shows the path of producing FlaxPP material. After collecting face masks, mask layers were separated and stacked with the flax fibre layers to form a composite laminate by compression moulding. Three different compositions: flax and polypropylene (PP), flax and virgin polypropylene sheets (vPP), glass and polypropylene were tested for vibration damping to evaluate their mechanical properties. Flax/PP and flax/vPP showed similar results of damping ratio, while the damping ratio of glass PP was reduced (18-35%) in comparison with Flax/PP. Hence, Flax/PP could be a sustainable alternative for applications where high-vibration damping is required (sports equipment, sound insulation panels, aerospace interiors). Also, FlaxPP composite could be recycled and used for less structural manufacturing purposes.[17]

Recycling bins for face masks should be established and after collecting they would have to be transported to a facility for 72 hours of decontamination, UV lighting or different chemical methods. This material has big potential thus environmental recovery.[17]

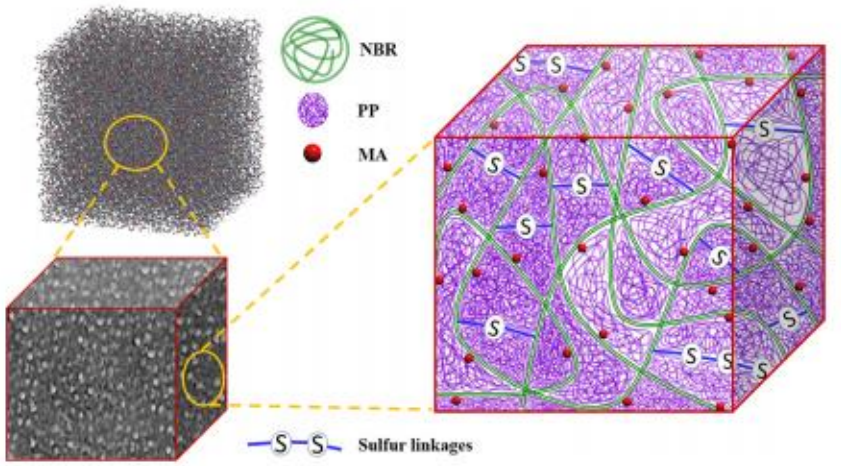

Figure 11. PP-NBR blend[24]

Polypropylene is non-polar, chemically inert, resistant to moisture and heat, also has a high resistivity and high abrasion resistance. On the other hand, NBR is polar rubber that is extraordinarily resistant and has high electrical conductivity and low-temperature flexibility. Non-polar polypropylene with polar acrylonitrile butadiene rubber (NBR) using maleic anhydride as the compatibilizer that acts like a bridge between PP and the NBR matrix (*Fig 11*) is used to produce PP-NBR which can be used for a wide variety of applications. Likewise, the PP-NBR blend is an engineering product made from used face masks with enhanced thermomechanical properties. This process is energy efficient as CO_2 footprint is low.[24]

Figure 12. Way of producing green concrete[5]

Fibers are added to concrete to increase its structural integrity because they control cracking due to plastic shrinkage and increase shatter resistance.[5] Face mask waste was found as a material that can be used in concrete, as it is shown in *Fig 12.* The fibers used in concrete to enhance properties are expensive and carbon fibers are harmful to the environment due to carbon emissions. Fibers from waste face masks represent a cheaper source of fibers which can improve the microstructure of mortar and concrete, durability and mechanical characteristics. In this study, the mask fibers were fibered in a cotton extracting machine and incorporated at 0.5, 1.0, 1.5 and 2.0% of concrete volume while crushed masks were crushed using a paper cutting machine and incorporated at 0.5% concrete volume. Masks, cement and sand crush were dry mixed and mixed with water. After that, concrete was cast in cylinders for each batch for 24 hours and then places in water at room temperature until test time. Hence, after 28 days of curing compressive strength was measured and it was concluded that the durability of concrete is improved with the addition of waste face masks at 0-1.5% fiber content. An optimum value of the addition of fiber content is 1% due to the durability and mechanical properties of concrete. Addition more than 2% results in a reduction of compressive strength because it is tough to distribute fibers evenly over that amount causing interlocking of fibers.[5]

Researchers applied a recycled polypropylene mask as a separator for aqueous rechargeable batteries (ARBs) through a reaction between polypropylene and fuming sulfuric acid. Through this process in 10 minutes, the hydrophobic surface of polypropylene is converted to hydrophilic with abundant -OH and -SO_3H groups. As a result, the material has better performance in comparison with conventional separators, such as glass fibre-based separators (GF). Although, GF has excellent wettability by aqueous electrolytes the cost and thickness (380 μm) have a negative impact on commercial ARBs. On the other hand, the filter layer of the face mask showed outstanding safety, electrochemical performance and allows facile electrolyte transport. In near future recycling the filter layer of face masks will be popular in fields of energy-transforming or bio-engineering.[2]

Three different compositions: filter and ear loop (a), filter and nose wire (b) and filter (c) were tested for mechanical properties to obtain a material with good mechanical properties that can be used in the production of heavy-duty kids chairs. After collecting, face masks were disinfected and melted by a conventional heat gun in a temperature range between 50-600 °C. Results from tensile strength showed the best results for composition made of filter and ear loop in comparison with others, because of the interaction between polypropylene from the filter and nylon from the ear loop.[1] This approach showed that it is possible to recycle polypropylene face masks by using simple experiment without expensive equipment or hazardous chemical solvent. Hence, this way of recycling is economical, green and represents an alternative in the production of various decoration and furniture products by reducing wood and raw polymer usage. In *Fig 13* kids chair produced from recycled polypropylene face masks (filter layer and ear loop) is presented.[1]

Figure 13. Kids chair produced from recycled polypropylene face masks (filter layer and ear loop)[1]

Also, there is a study that observed properties of the 3D printing extrusion to the recycled disposable face masks. The ratio of 30% of talc and (35 and 50 w%) filtering material was identified as suitable for the printing process. The material from the mask shows similar behavior to a commercial material including the same amount of talc. Accordingly, tests showed a good stiffness and strength that can be compared to the commercial 3D printable material and can be used in industry. This study reports the first case where disposable filtering masks were used to produce filaments for the production of extruded 3D printed objects, as the beginning of producing new formulations which will valorize highly impacting waste.[25]

Pyrolysis is an alternative method to recycle face masks and does not require prior waste separation. Due to the high polypropylene content in single-use face masks, these could be used in the production of great yields of liquid and gas fuels. [22]

1.3.1. Mechanical recycling

Mechanical recycling is a process that involves four steps: thermochemical disinfection treatment, elimination of metal and ear loop, grinding, extrusion and injection. By using this method, it would be possible to reduce the waste of single-use face masks and the environmental pollution that Covid-19 caused.[26] The mechanical properties of recycled polypropylene make it useful to obtain storage bins and flower pots where technical specifications are not critical.

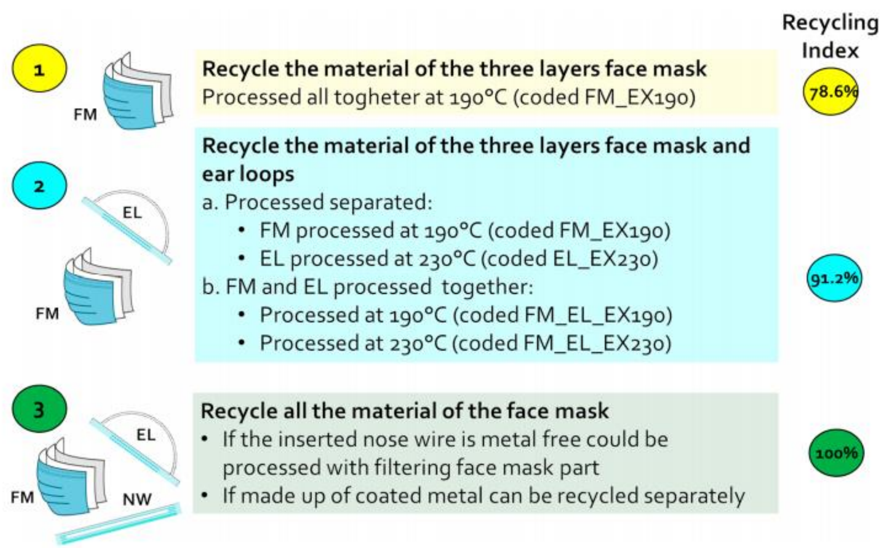

Figure 14. Three different recycling methods[27]

In the first method nose wire and ear loop were separated manually and then the face mask was recycled, with a recycling index of 78.6%. In the second method, the ear loop was previously separated from the mask and then recycled (*Fig 14(2a)*) or (*Fig 14(2b)*) ear loop was recycled with the face mask without a drying process. With this method recycling index increases to 91.2%. In the third method, the nose wire was separated and recycled with metal and face mask separately with a recycling index of 100%.[27] Face masks were separated manually (face mask, ear loop, nose wire), grinded with the aid of scissors and extruded in a co-rotating twin screw micro extruder at 190 °C (the temperature at which polypropylene is melted) and 230 °C (the temperature at which polyamide 6 from ear loop is in a molten state) under the chosen strategy. After extrusion, films were obtained by using a hot compression moulding press. Thermal, mechanical, rheological, chemical and morphological analyses were conducted and it was concluded that face masks can be mechanically recycled.[27]

Recycling with the first method (*Figure 14(1)*) resulted in low complex viscosity and elongation at break in comparison with polypropylene. In that case, recycled face masks can be exploited in a mixture with first use of polypropylene with higher viscosity for direct use or filler from waste can be added given the low complex viscosity of the recycled mask material. The second method (*Figure 14(2a)*) includes the recycling of ear loops as well and polyamide 6 recycled from ear loops showed complex viscosity like polyamide 6 and tensile performances reveal properties <50% of a common polyamide 6. After recycling this type of material can be exploited in injection moulding for applications where high mechanical characteristics are not required. In the method (*Figure 14(2b)*) ear loop is recycled with a face mask and different types of materials are mixed which manifests in the worst mechanical results.[27] It can be concluded that methods 1 and 2a (*Fig 14*) have the potential for industrial exploitation but need more research for future developments.[27]

Plastic extruding companies reprocess plastic waste through a twin-screw extrusion process. But often the problem with face masks is heterogeneous composition. That is why technical studies must be accompanied by economic analysis and scale-up prospects to achieve a better understanding of their implementation cost and viability.[22] By this research, it is suggested that extruded face mask material could be directly recycled by injection moulding or complement its mechanical properties with fillers from agro-industrial wastes.[22]

1.4. Biodegradable masks

Polypropylene in a face mask can be replaced with bioplastic and biodegradable polymers derived from biological substances which could degrade through natural biological processes into non-toxic gases and carbonaceous soil over time.[4] Also, in *Fig 15*(*a)* and *Fig15(b)* can be seen similarities in the woven technology used in the filter layer of single-use face mask and plant fibre filter.[16] Natural and synthetic biodegradable polymers are used to develop biodegradable fibrous material for potential biodegradable face masks. Synthetic fibres are easier to process with tailored mechanical properties, while natural fibres have better biodegradability and biocompatibility.[4] Production of biodegradable masks requires precisely defined standards such as water resistance, elasticity and filtering properties. The production of polypropylene face masks releases 59 g CO_2 per single mask, while biodegradable plastic reduces the CO_2 emission by 30% to 70%.

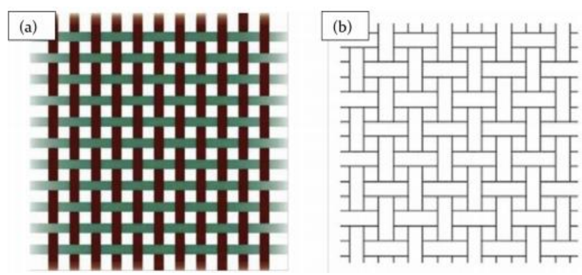

Figure 15. Woven technology in a) filter layer of single-use face mask and b) plant fibre[16]

Natural fibres such as coffee, cactus, hemp, straw, banana, sisal, avocado, bamboo, sugar cane and lotus can be used for the production of a biodegradable mask. But not only raw natural fibres can be used in production, there is also a possibility for natural fibre waste such as tea leaf waste which contains polypropylene and enhances the properties of polyacrylic acid in bioplastics. Today on the market in limited countries hemp fibre mask, coffee-based face mask and sugar cane waste mask are available and they are obtained with 99.99% dual antibacterial technology and improved filtering effectiveness.[16, 22] Biodegradable and biobased polymers are more expensive than conventional plastics (PP, PE, PET), so the production of biodegradable face masks is limited. Although biodegradable face masks made from bioplastics claim to be degradable, there are still restrictions cause of the uncertainty about the complete degradable nature of the biodegradable face masks. Scientists should examine the profitability of such a project.[16] However, biodegradation requires industrial scale composter and appropriate infrastructure for biowaste management for some plastics and that is the reason why there is a barrier created for developing countries with poor solid waste management.[16, 22]

Table 2. Bio-based materials for single-use face masks [4]

Bio-based media	Structure and materials	Applications
Protein	Keratin / polyamide 6 nanofiber Electrospun sericin nanofibrous mats Silk nanofibers Gluten nanofiber Soy protein isolate/ PVA hybrid nanofiber Nanomembrane lyocell fibers	Water and air filtration Air filtration mask Air filtration mask Face mask
Cellulose	Cellulose non-woven layers Cellulose acetate nanofibers 3-ply cotton-PLA-cotton layered Fungal hyphae and cellulose fibers (wood and hemp) Banana stem fiber Non-woven cellulosic fiber	Surgical face mask Air filtration mask Face mask Alternative to syntheric melt and spun-blown materials for PPE Face mask Face mask
Chitosan	Nanofibrous chitosan non-woven	Water and air filtration
Poly lactic acid	Poly lactic acid fibrous membranes 3D printed and electrospun polylactic acid	Air filtration Face mask filter
Gelatin	Gelatin / β-cyclodextrin composite nanofiber	Respiratory filter

There is significant interest in material research and development of complete or partial reusable fibrous face masks based on biodegradable materials to find a sustainable solution to the environmental problem of waste management. *Table 2* represents materials that have been used in several commercially available biodegradable filtration media for face masks.[4] Also, there is a possibility of developing environmentally friendly next-generation face masks based on multifunctional membranes in which the presence of plasmonic nanoparticles and nanoclusters enables the chemically destruction of pathogens. Cu nanoclusters are used for their chemically driven intrinsic antibacterial and antiviral activities, while Au nanoparticles are effective photo converters for harnessing synergistically assisted photothermal disinfection.[4]

2. EXPERIMENTAL PART

2.1. Material

Single-use face masks used in this paper were purchased from Guangdog annuochen medical supplies c. Ltd. Total weight of one single-use face mask was 3.0442 g (average of 3 single-use face masks). *Fig 16* shows a single-use face mask used in this paper.

Figure 16. Single-use face mask

Firstly, components of the single-use face mask were manually separated: ear loop (*Fig17(a)*), metal material in the nose part (*Fig17(b)*) and face filter (*Fig17(c)*). For analysis, only a face filter was used. *Fig 18* shows separated layers of face filter: inner layer a), filter layer b) and outer layer c).

Figure 17. Single-use face mask components – a) metal material, b) ear loop, c) face filter

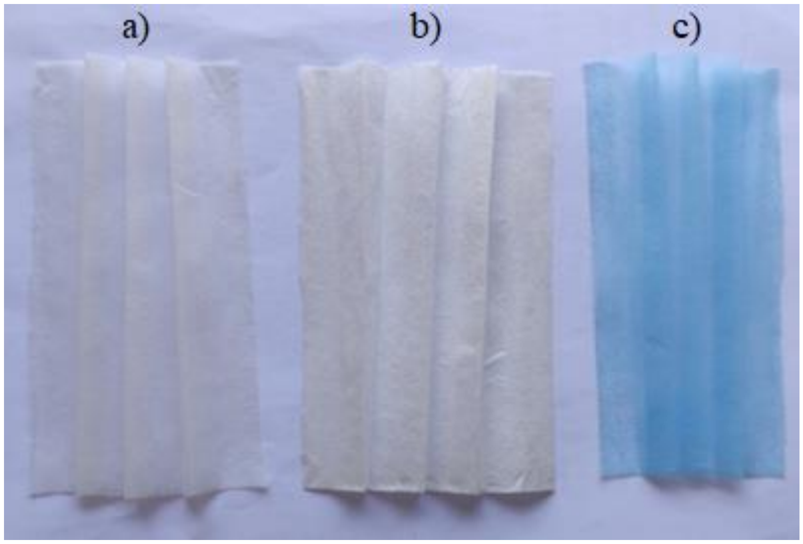

Figure 18. Separated layers of single-use face mask: a) inner layer, b) filter layer, c) outer layer

2.2. Mechanical recycling

As has been described above, non-used single-use face masks were manually separated from each layer (*Fig 17*), and only a face filter (*Fig 17(c)*) was recycled in this paper. Each layer that composes a single-use face mask was manually separated and grinded by scissors. Mechanical recycling of the single-use face mask was done by a laboratory single-screw extruder shown in *Fig 19* at a temperature of 160 °C and a screw speed of 160 rpm. *Fig 20* shows a sample obtained with the material from a single-use face mask for mechanical characterization. It can be seen that after recycling the colour of the sample is identical to the base material; no degradation occurred.

Figure 19. Laboratory single-screw extruder Dynisco (Qualitest, USA)

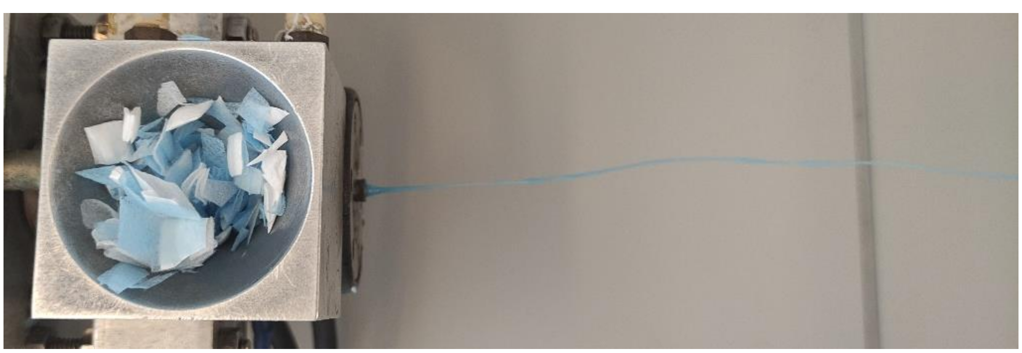

Figure 20. Recycled single-use face mask

2.3. Material characterization

Single-use face mask (*Fig17(c)*), separate layers of single-use face mask (*Fig 18*) and recycled single-use face mask (*Fig 20*) were characterised by FTIR, DSC and TG.

Results of characterisation of separate layers are described in chapter 2.3.1, 2.3.2 and 2.3.3. While, results of characterisation of the single-use face mask and recycled single-use face mask are described in chapter 3.3.1, 3.3.2 and 3.3.3.

2.3.1. Fourier-transform infrared spectroscopy

Fourier transform infrared spectroscopy spectra were recorded on Perkin Elmer Spectrum Two FTIR spectrometer by the Universal Attenuated Total Reflectance (UATR) technique with diamond reflection crystal. The spectra were collected in 10 scans at a resolution of 4 cm^{-1} and in the range of 4000–450 cm^{-1} at room temperature. *Fig 21* shows PerkinElmer Spectrometer Spectrum Two.

Figure 21. PerkinElmer Spectrometer Spectrum Two

2.3.2. Differential scanning calorimetry

Differential scanning calorimetry is the most widely used thermal analysis technique in polymer science and it is economical because it requires only milligram quantities for the measurements.[28] With this technique the amount of heat absorbed or evolved by a sample in comparison with a reference during a specific thermal transition can be measured. The changes in thermal properties of the sample can be determined as a function of time (isothermally) or temperature (at a constant heating or cooling rate).[29]

The DSC curve shows the change in heat flow as a function of temperature, as is shown in *Fig 22* in which all possible thermal changes that may occur in the sample are recognized. The features of the DSC curve are shown in *Table 3* in accordance with international standards, HRN EN ISO 11357-2: 2013 and HRN EN ISO 11357-3: 2011.[30]

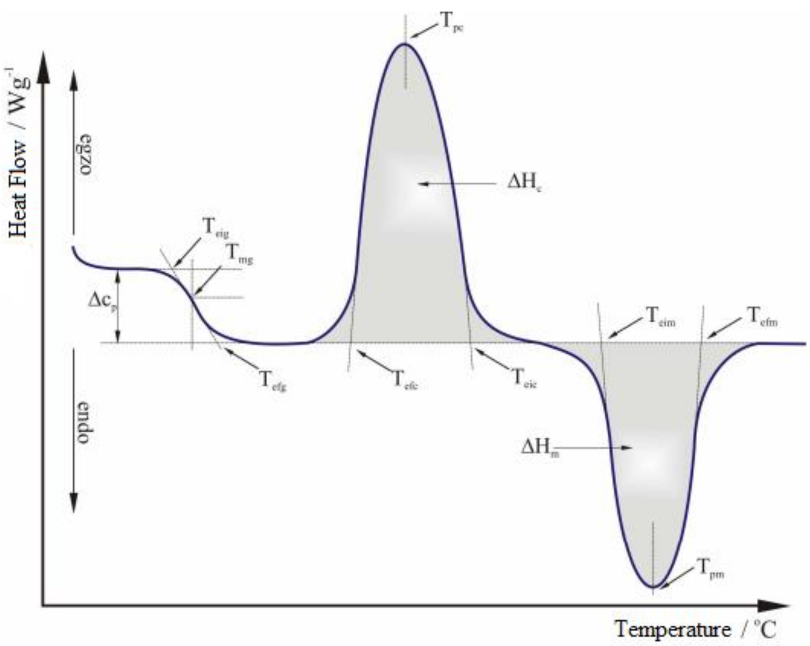

Figure 22. DSC curve[30]

Table 3. Features of DSC curve: norms HRN EN ISO 11357-2: 2013 and HRN EN ISO 11357-3: 2011[30]

Feature	Label
Glass transition	T_{eig} – extrapolated initial temperature / °C
	T_{mg} – midpoint temperature / °C
	T_{efg} – extrapolated final temperature / °C
	Δc_p – change in the specific capacity of the glass transition / J g^{-1} $°C^{-1}$
Melting	T_{eim} – extrapolated initial melting temperature / °C
	T_{pm} – minimum melting temperature / °C
	T_{efm} – extrapolated final melting temperature / °C
	ΔH_m – melting heat / J g^{-1}
Crystallization	T_{eic} – extrapolated initial crystallization temperature / °C
	T_{pc} – temperature in maximum crystallization / °C
	T_{efc} – extrapolated final crystallization temperature / °C
	ΔH_c – heat of crystallization / J g^{-1}

The degree of crystallinity (%) is calculated using the following expression:

$$X_c(\%) = \frac{\Delta H_m}{\Delta H_m^0} \cdot 100$$

In this expression:

ΔH_m – heat of melting of the sample (J g^{-1}) is measured by DSC

ΔH_m^0 – heat of melting of 100% crystalline PP (J g^{-1}) – 207 J g^{-1}, the reference value for the heat of melting. [34]

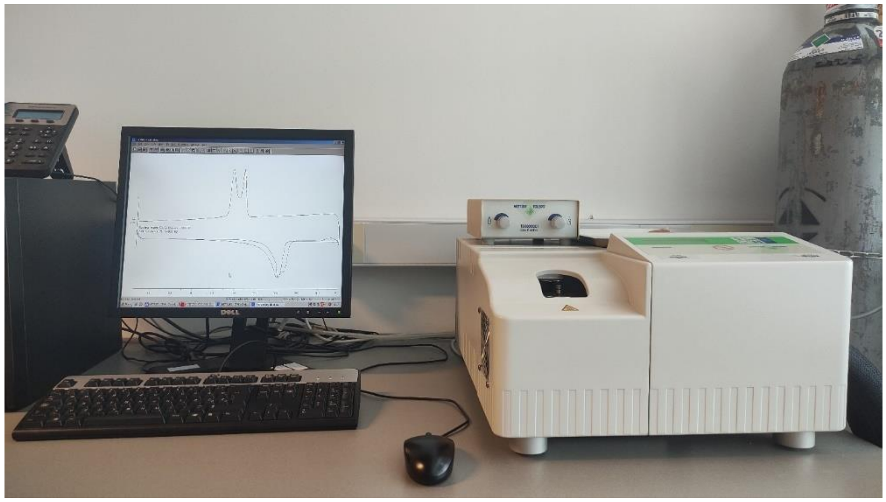

***Figure 23**.* Mettler Toledo DSC 823^e

Thermal properties of single-use face masks were investigated by Mettler Toledo DSC 823^e calorimeter (*Fig 23*) in a nitrogen atmosphere (30 cm^3 min^{-1}). Samples of approximately 10 mg were heated at a rate of 10 °C min^{-1} from 25 to 220 °C, cooled at the same rate to 25 °C, and reheated to 220 °C. The instrument was stabilized for one hour before the beginning of the measurements. The samples were pressed in aluminium pans. The first heating scan curve represents the influence of the thermal history of the sample. The cooling scan curve presents the crystallization from the melt of the samples after the thermal history is removed.

2.3.3. Thermogravimetric analysis

Thermogravimetric analysis (TG) provides quantitative measurement of the mass change without information of the nature of the material lost, but thermal stability, composition and extent of cure can be characterized.[31] Green curve (TG) in *Fig 24* is a result of dynamic thermogravimetric degradation and represents the change in the mass of the sample as a function of temperature. The orange curve represents the derivative TG curve, known as DTG curve which represents the rate of mass loss with temperature. The features of TG and DTG curves are shown in *Table 4.*

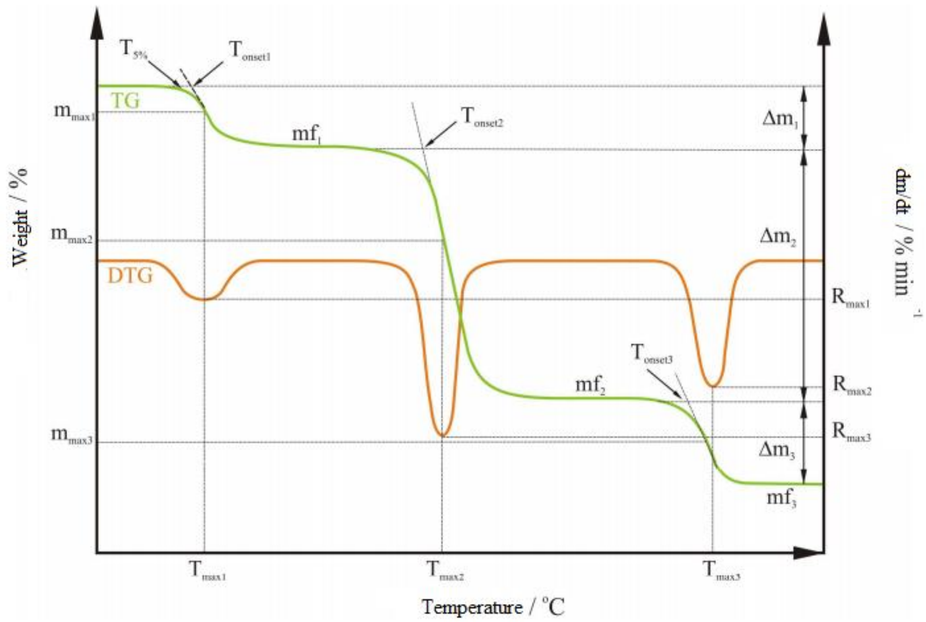

Figure 24. TG and DTG curves[30]

Table 4. Characteristics of TG and DTG curves[30]

Feature	Description
T_{onset} / °C	the onset temperature
$T_{5\%}$ / °C	the temperature at which the sample loses 5% of its initial mass
T_{max} / °C	temperature at the maximum degradation rate, corresponds to the minimum of the DTG curve
R_{max} / % min^{-1}	maximum decomposition speed
Δm_f / %	the mass loss at a particular degradation stage
m_f / %	the remaining mass at the end of the thermal decomposition process

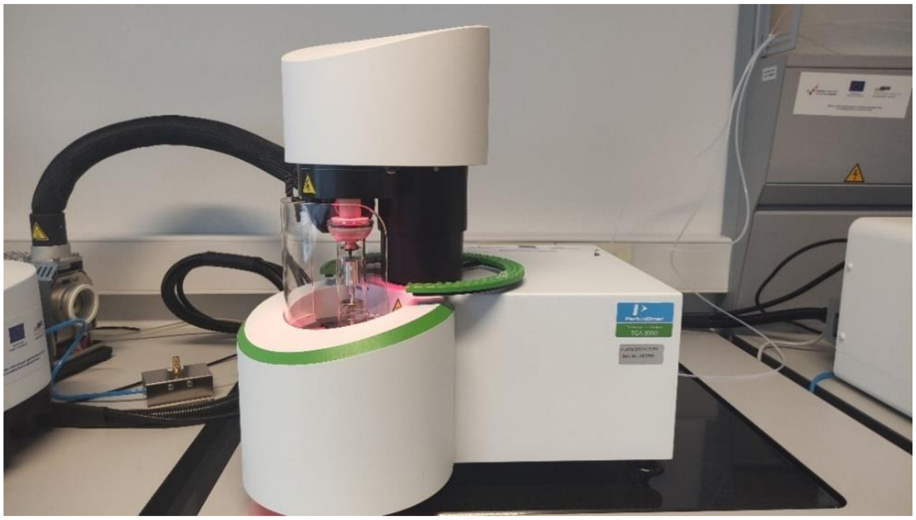

Figure 25. PerkinElmer Thermogravimetric Analyzer TGA 8000

Thermogravimetric measurements were performed by PerkinElmer TGA 8000 Analyzer (*Fig 25*) at heating rate of 10 °C min^{-1} in a temperature range 30-600 °C in nitrogen (40 cm^3 min^{-1}). Mass of the samples used was approximately 10 mg.

3. RESULTS AND DISCUSSION

3.1. Characterization of single-use face mask material

3.1.1. Fourier-transform infrared spectroscopy

In order to obtain data on the main material that single-use face masks are made of, commercial single-use face masks shown in *Fig 17(c)* were analysed by UATR-FTIR. The FTIR spectra of the outer (blue), the filter (red) and the inner (green) layer were obtained separately (*Fig 26–28*) and analysis of the characteristics peaks was conducted in order to identify the main polymer that composes each layer. In order to identify the main polymer that composes each layer, the assignments of the most representative peaks found in the spectra are shown in *Table 5* and compared with available literature. As a result, it was confirmed that the inner layer, filter layer and outer layer have the same characteristic peaks as polypropylene FTIR spectra. Polypropylene spectra contain at least four vibration bands associated with the CH_2/CH_3 stretching, while polyethylene contains two vibration bands associated with this mode of vibration and the relative intensity of these bands are different as well.[26] The FTIR spectrum showed large bands at 2950 cm^{-1} which is related to CH_3 asymmetric and symmetric stretching vibrations. Also, the band at 2920 cm^{-1} is connected to CH_2 asymmetric and symmetric stretching vibrations. Intense bands at 1456 cm^{-1} and 1376 cm^{-1} are related to CH_3 symmetrical bending. The band at 1166 cm^{-1} is attributed to C – H wagging vibrations and CH_3 asymmetric rocking.[31, 32]Also, the bands at 996 cm^{-1} and 973 cm^{-1} are connected to CH_3 rocking vibrations. The band at 840 cm^{-1} is connected with rocking C – H bond. The band at 808 cm^{-1} is related to stretching C – C bond.[33]

Hence, the main polymer from which each layer of face mask is made is definitely polypropylene.

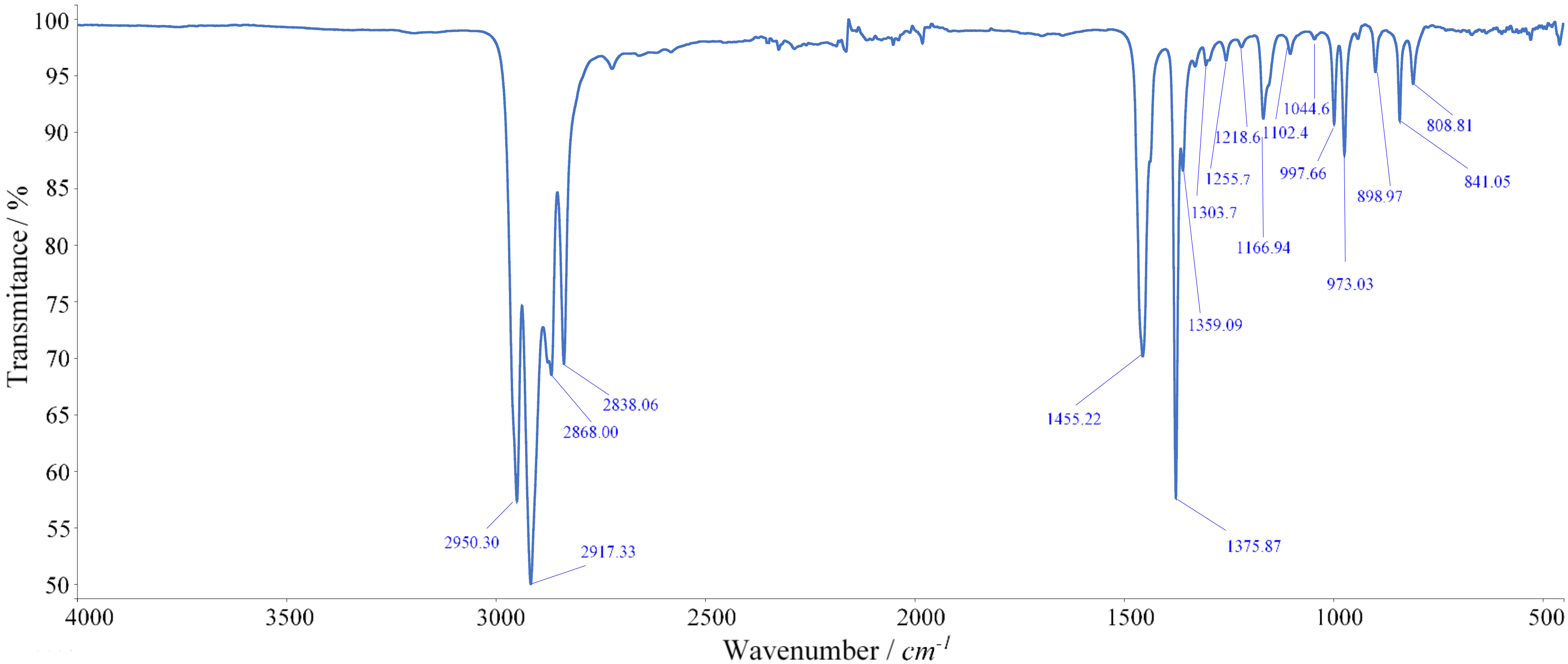

Figure 26. FTIR spectrum of outer layer

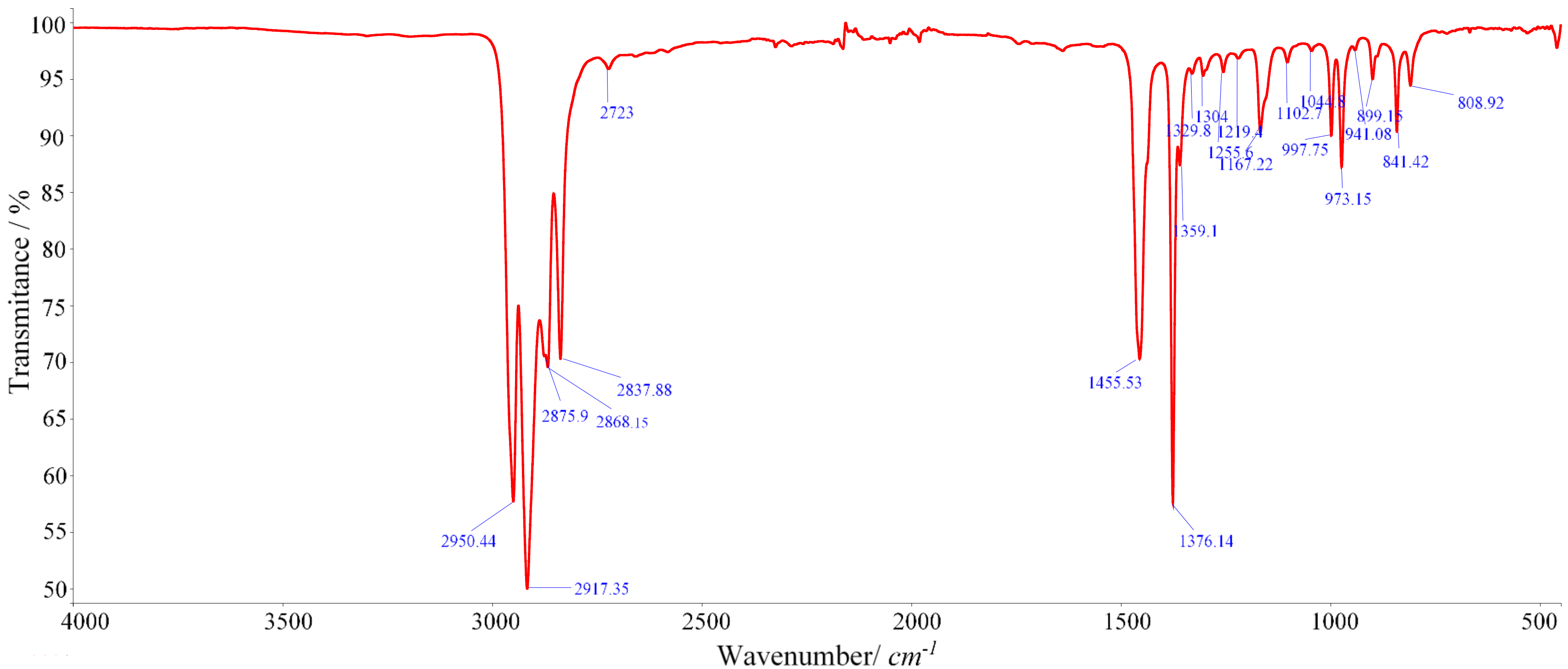

Figure 27. FTIR spectrum of filter layer

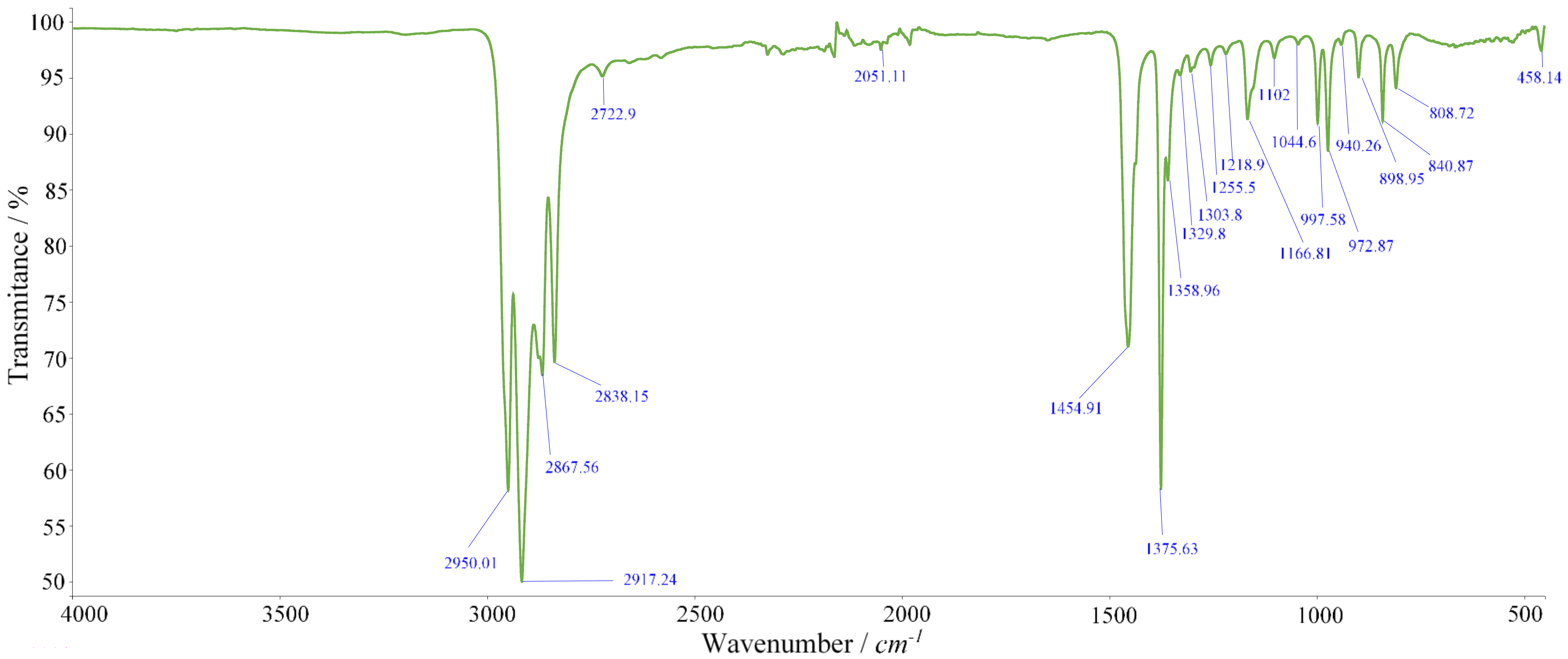

Figure 28. FTIR spectrum of inner layer

Table 5. FTIR peak assignments of outer layer, filter layer, inner layer and recycled single-use face mask

Outer layer	Filter layer	Inner layer	Recycled single-use face mask	Literature	Vibration type	Assignment	Refs.
	Wavenumber / cm^{-1}						
2950	2950	2950	2951	2950	Asymmetrical and symmetrical stretching	CH_3	[30]
2917	2917	2917	2917	2920	Asymmetrical and symmetrical stretching	CH_2	[30]
2868	2868	2868	2876	2870	Asymmetrical and symmetrical stretching	CH_3	[31]
2838	2838	2838	2837	2840	Asymmetrical and symmetrical stretching	CH_2	[30]
1455	1456	1455	1455	1456	Asymmetrical and symmetrical bending (in plane)	CH_2	[30, 31]
1376	1376	1376	1376	1376	Asymmetrical and symmetrical bending (in plane)	CH_3	[30, 31]
1167	1167	1167	1166	1166	Wagging Rocking	C-H CH_3	[31]
998	998	998	996	996	Rocking	CH_3	[31]
973	973	973	973	973	Rocking	CH_3	[31]
841	841	841	840	840	Rocking	C-H	[31]
809	809	809	808	808	Stretching	C – C	[31]

3.1.2. Differential scanning calorimetry

Fig 29-31 shows DSC curves of the inner layer, filter layer and outer layer. The first heating scan is marked (red), cooling scan (blue) and the second heating scan (green). While, *Fig 32-34* shows a comparison of each thermal transition. *Table 6* shows thermal transition parameters determined from DSC curves.

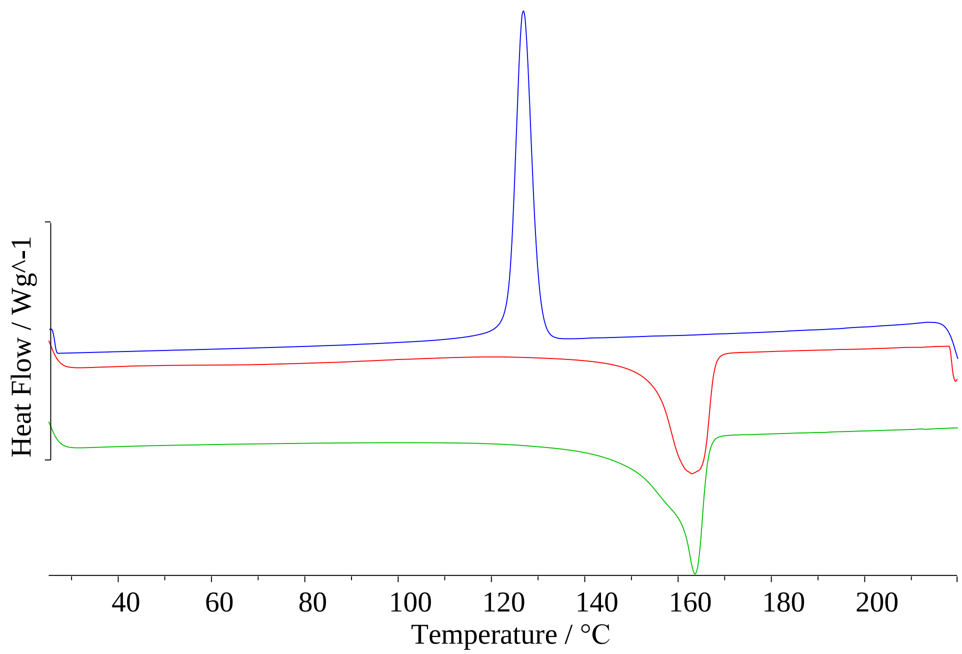

Figure 29. DSC curve of outer layer

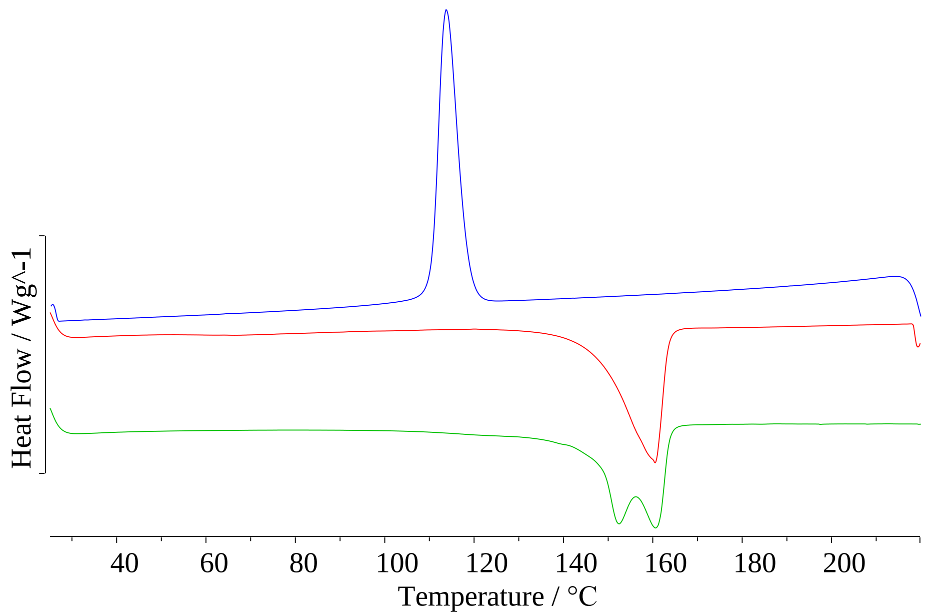

Figure 30. DSC curve of filter layer

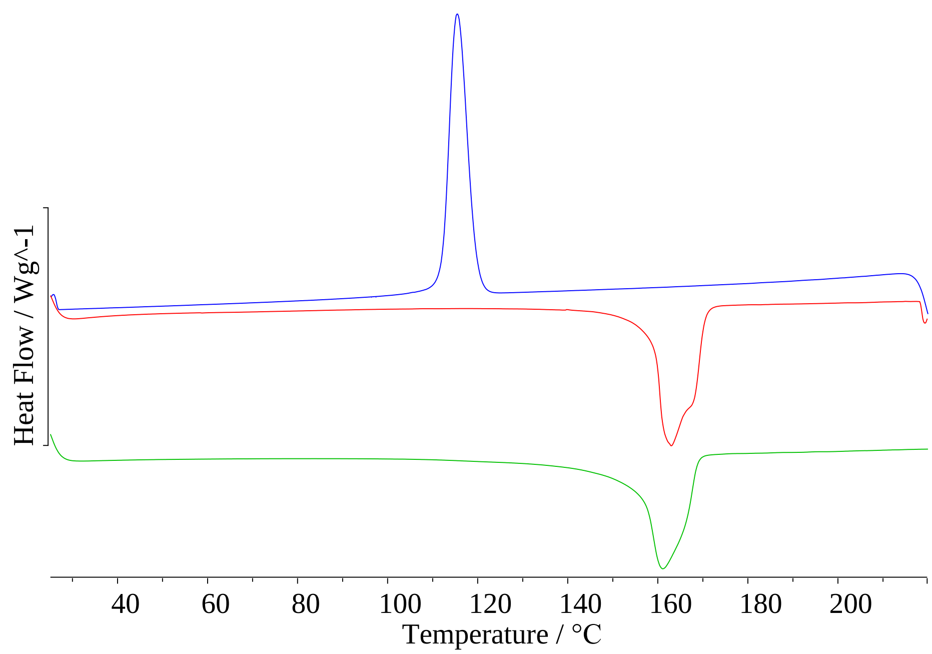

Figure 31. DSC curve of inner layer

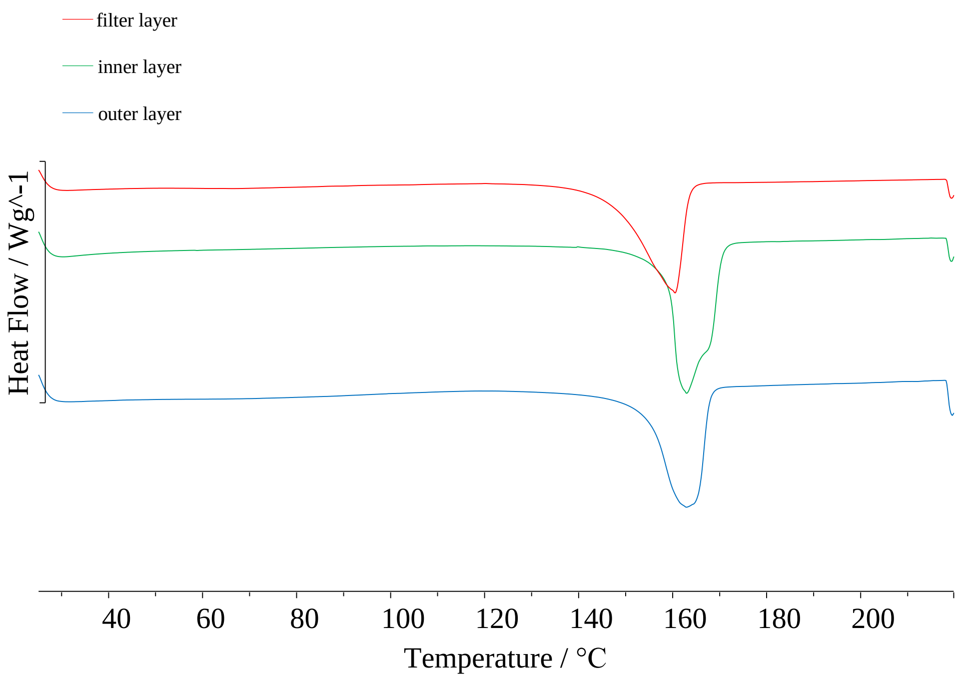

Figure 32. DSC curves of the first heating scan for outer layer, filter layer and inner layer

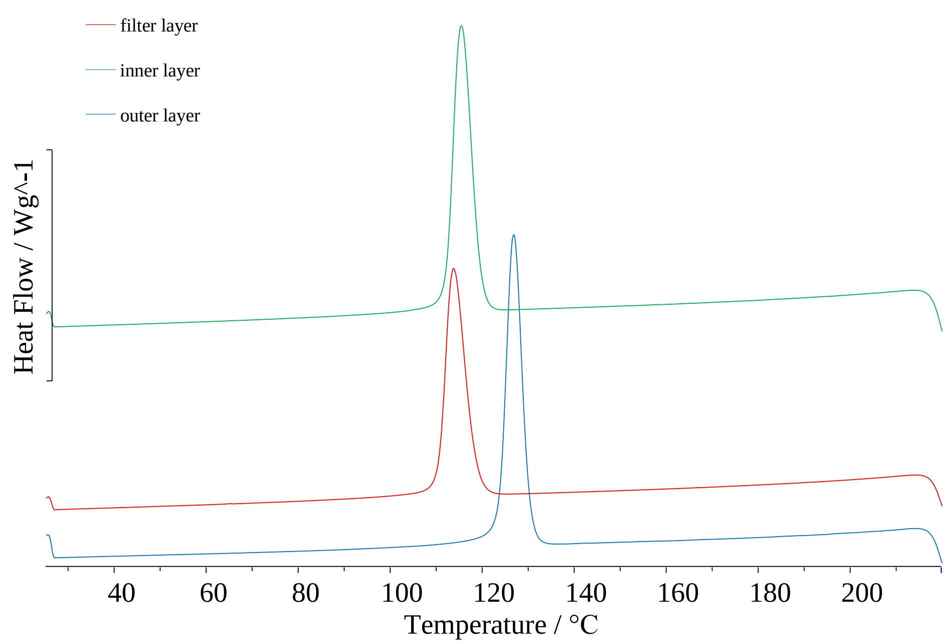

Figure 33. DSC curves of cooling scan for outer layer, filter layer and inner layer

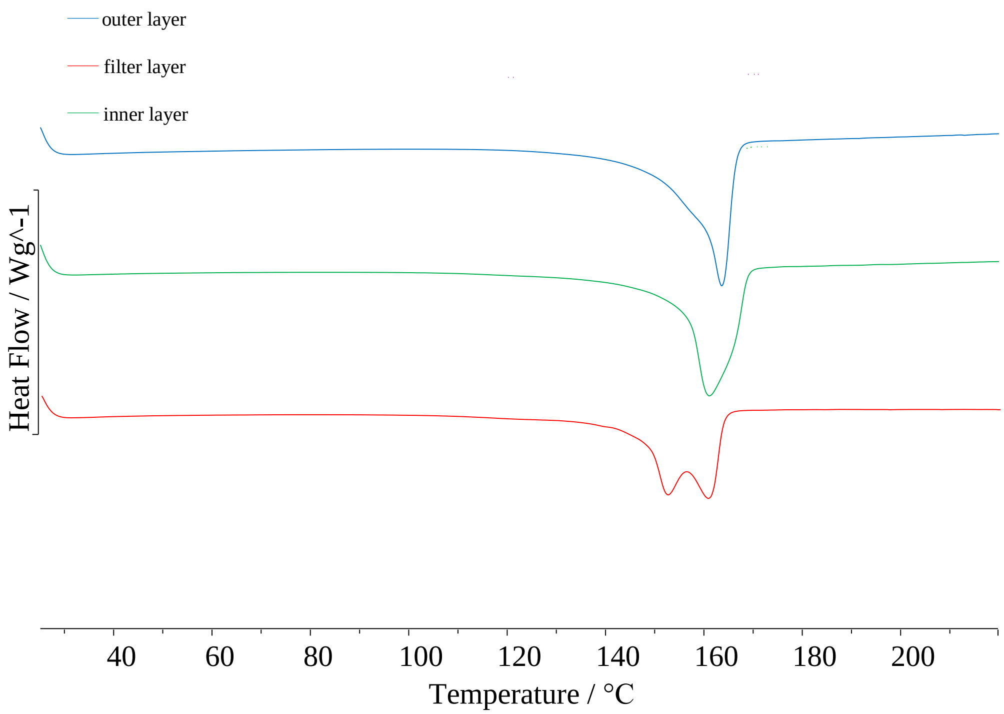

Figure 34. DSC curves of the second heating scan for outer layer, filter layer and inner layer

Table 6. Thermal transition parameters of outer layer, filter layer and inner layer

Sample	1st Heating				Cooling				2nd Heating				
	T_{eim} /°C	T_{pm} /°C	T_{efm} /°C	$-\Delta H_m$ / J g^{-1}	T_{eic} /°C	T_{pc} /°C	T_{efc} /°C	$-\Delta H_c$ / J g^{-1}	T_{eim} /°C	T_{pm} /°C	T_{efm} /°C	$-\Delta H_m$ / J g^{-1}	X_c / %
Outer layer	154	163	167	82.92	130	127	124	89.90	158	163	167	89.88	43.42
Filter layer	148	160	163	71.01	119	114	111	77.44	147	152 161	163	80.2	38.7
Inner layer	159	163	170	88.40	120	116	112	92.49	156	161	169	93.87	45.35

The thermal characteristics of investigated samples were determined by differential scanning calorimetry in accordance with the norms HRN EN 11357-2: 2013 and HRN EN 11357-2: 2011. DSC curves of each layer are normalized (reduced to a mass of 1 g in order to compare thermal effects).

DSC curves of each layer *(Fig 29, 30, 31)* showed an endothermic peak in the temperature region of 160-163 °C. These temperatures are comparable with the isotactic polypropylene melting point and in accordance with results of FTIR analysis.[34]

On the heating curve (first heating) of outer layer endothermic peak at 163 °C is related to melting of the crystalline phase of polypropylene. The melting temperature of outer layer is 154 °C (T_{eim}). While the melting temperature of inner layer is 159 °C (T_{eim}). The melting temperature of filter layer is 148 °C (T_{eim}). Inner layer (88.40 J g^{-1}) has the highest value of melting enthalpy, then outer layer (82.92 J g^{-1}), while filter layer (71.01 J g^{-1}) has the lowest value.

On the cooling curve of outer layer exothermic peak at 127 °C is related to crystallization of polypropylene. For outer layer at 130 °C (T_{eic}), inner layer at 120 °C (T_{eic}), filter layer at 119 °C (T_{eic}). Inner layer (92.49 J g^{-1}) has the highest value of crystallization enthalpy, then outer layer (89.90 J g^{-1}), while filter layer (77.44 J g^{-1}) has the lowest value.

On the second heating curve of outer layer melting temperature is 158 °C (T_{eim}), for inner layer is 156 °C (T_{eim}). On the other hand, filter layer showed two melting peaks. After the filter layer is melted, cooled and reheated, two melting peaks are obtained at 152 and 161 °C (T_{pm}). The melting of filter layer begins at 147 °C with an overall melting enthalpy of 80.2 J g^{-1} which is significantly lower than the value of the other two layers, which is attributable to the differences in thermal histories.

Polypropylene can exist in several crystal modifications depending on crystallization conditions and isotacticity and the monoclinic α-modification is predominant in a structure formed during slow cooling from the melt.[35] In the available literature, the source of double-peak shapes is related to processes involving α crystals only. More precisely, double melting endotherms have been attributed to transitions between different modifications of α crystal form. Two limiting structures, α_1 and α_2 forms have been postulated from the α crystal form, differing in degrees of disorder in the up- and down- positioning of the chains. Also, researchers proposed that a 'continuum' of different structures from the limiting disorder modification α_1 to the limiting ordered modification α_2 could exist instead of only two limiting forms.[36]

For α-PP the lower temperature endothermic peak is related to the melting of the crystals formed during the non-isothermal crystallization, while the higher temperature is caused by the melting of the crystals after the recrystallization/reorganisation, which have higher stability and perfection.[37]

3.1.3. Thermogravimetric analysis

Fig 35-37 shows TG and DTG curve of the inner layer, filter layer and outer layer, while (*Fig 38(a)*) shows TG curve of each layer and (*Fig 38(b)*) DTG curve of each layer. *Table 7* shows parameters determined from TG and DTG curves.

The main mechanism of thermal decomposition of polypropylene is the statistical breaking of polymer chains followed by the process of radical transfer with main degradation products pentane, 2-methyl-1-pentene and 2,4-dimethyl-1-heptene. Degradation process of polypropylene is manifested by the appearance of only one peak on the DTG curve.[38]

Degradation of inner layer and outer layer is carried out in a single stage, presenting the decomposition at an approximate temperature range 370-490 °C, which is connected with degradation of polypropylene. On the other hand, degradation of the filter layer is carried out in two stages: 300-370 °C and 370-490 °C as shown in Fig 36. This is connected with two melting peaks obtained at 152 and 161 °C (T_{pm}) in DSC analysis. For the first peak of filter layer in the temperature range of 300-370 °C it can be seen from Table 7 that the remaining mass at the end of the thermal decomposition process is 96%, while for the second peak in the temperature range of 370-490 °C all samples degraded completely, without leaving any significant residue.

Hence, no exact explanation for this peak in the temperature range of 300-370 °C cannot be provided only by TG analysis.

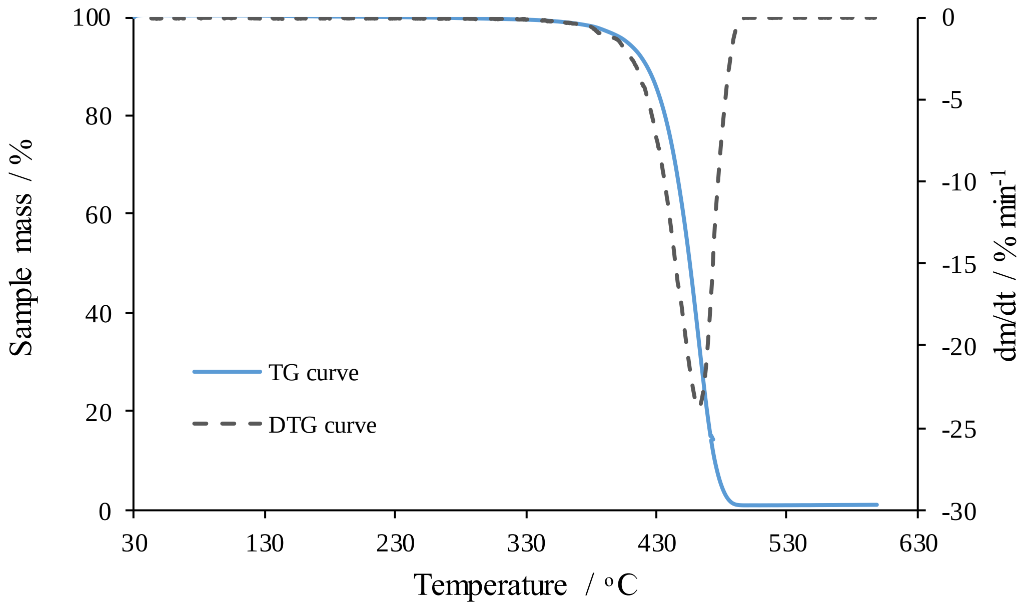

Figure 35. TG and DTG curve of outer layer

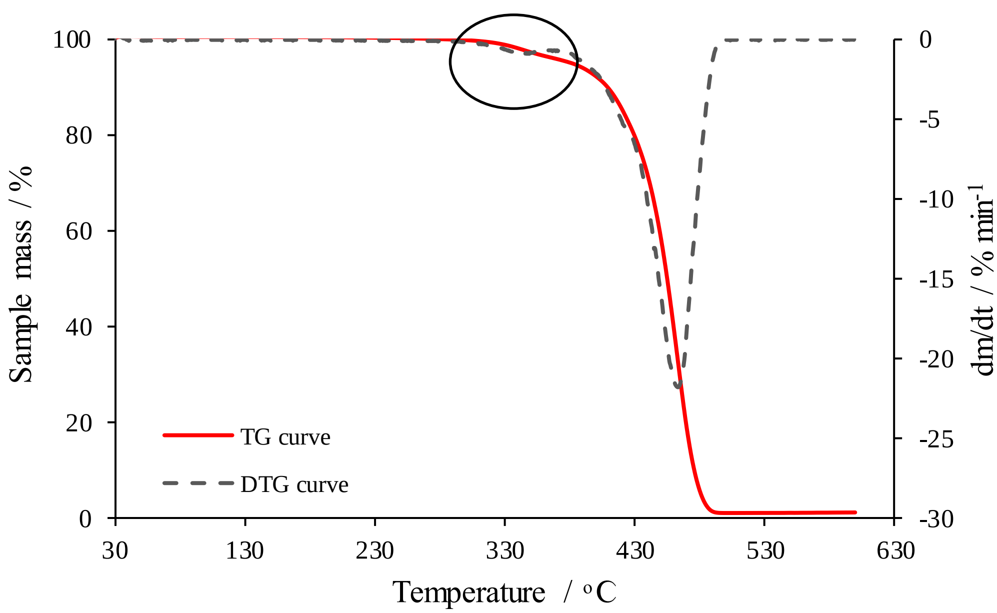

Figure 36. TG and DTG curve of filter layer

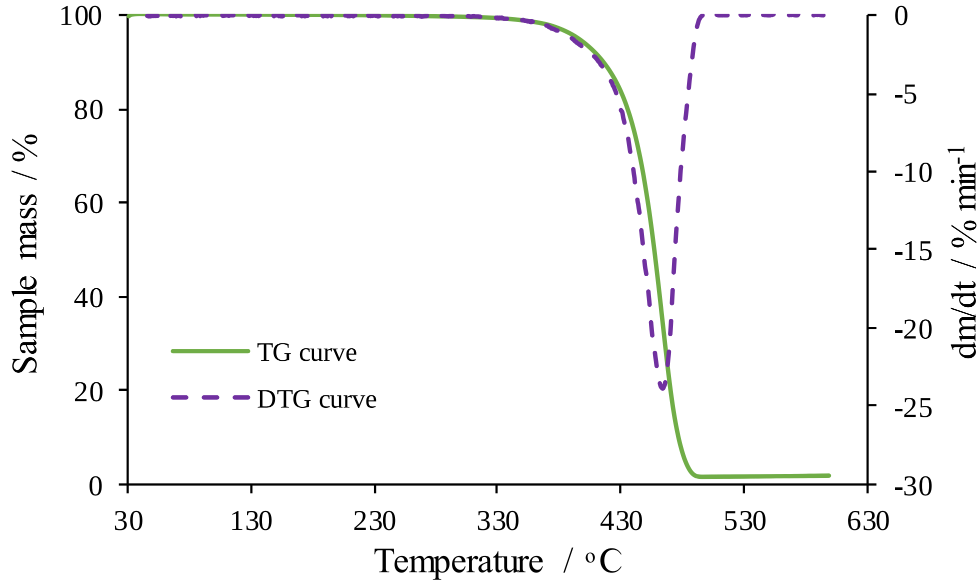

Figure 37. TG and DTG curve of inner layer

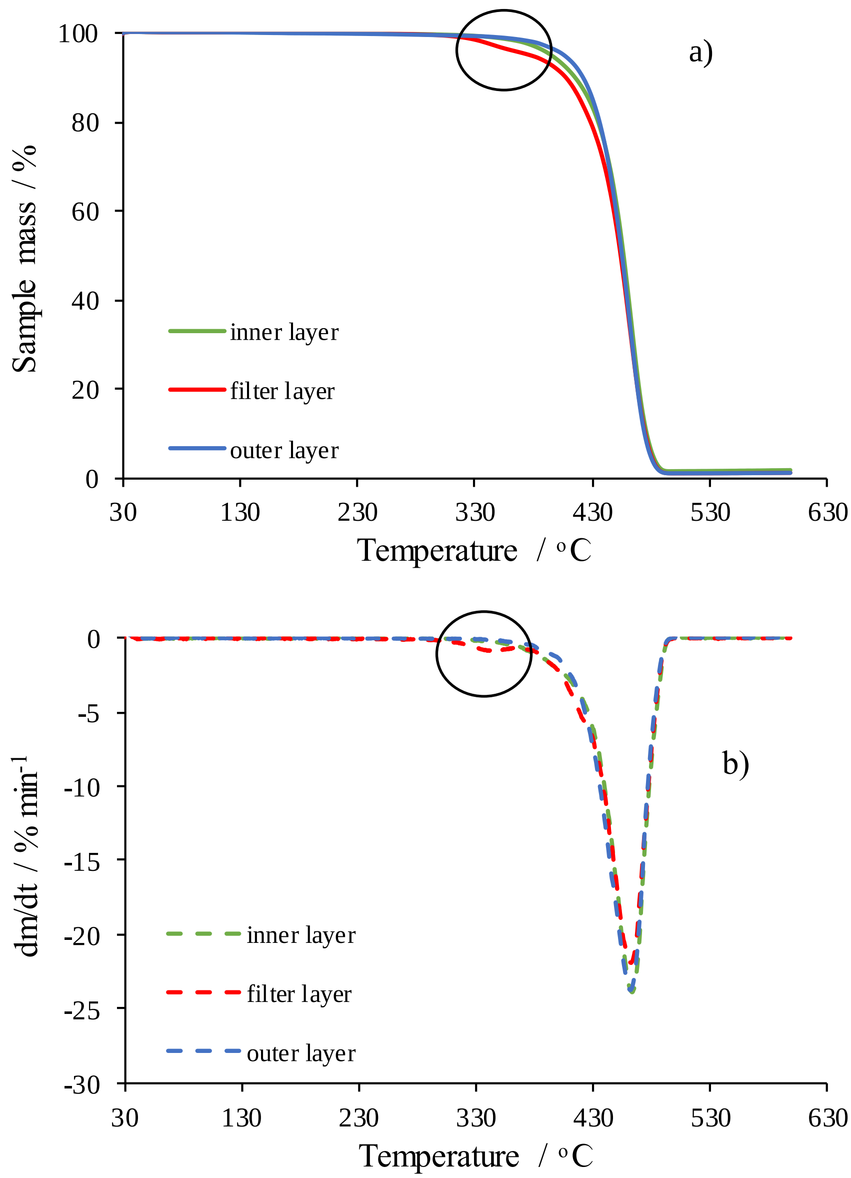

Figure 38. TG (a)) and DTG (b)) curves of outer layer, filter layer and inner layer

Table 7. TG and DTG thermal characteristics of outer layer, filter layer and inner layer

Sample	$T_{5\%}$ / °C	T_{onset} / °C	T_{max} / °C	R_{max} / % min^{-1}	Δm / %	m_f / %
Outer layer	407	433	462	23.8	98.8	1.1
Filter layer	365	315	341	0.9	4.3	96
	/	434	465	21.8	94.7	1.3
Inner layer	397	435	464	23.9	98.4	1.7

3.2. Characterization of recycled single-use face mask

3.2.1. Fourier-transform infrared spectroscopy

Recycled single-use face mask was analysed by UATR-FTIR and the result is shown in *Fig 39*. Comparison of FTIR spectra of a single-use face mask (each layer) with FTIR spectra of a recycled single-use face mask is shown in *Fig 40.*

In comparison, FTIR spectra of the outer layer, filter layer and inner layer with FTIR spectra of a recycled single-use face mask, there are no changes in the intensity, shape, and position of the characteristic peaks. Therefore, results of the FTIR analysis have confirmed that mechanical recycling did not affect the structure of a polypropylene single-use face mask. Recycled single-use face mask shows peaks at the same wavenumber (*Table 5*) as described in chapter 3.1.1.

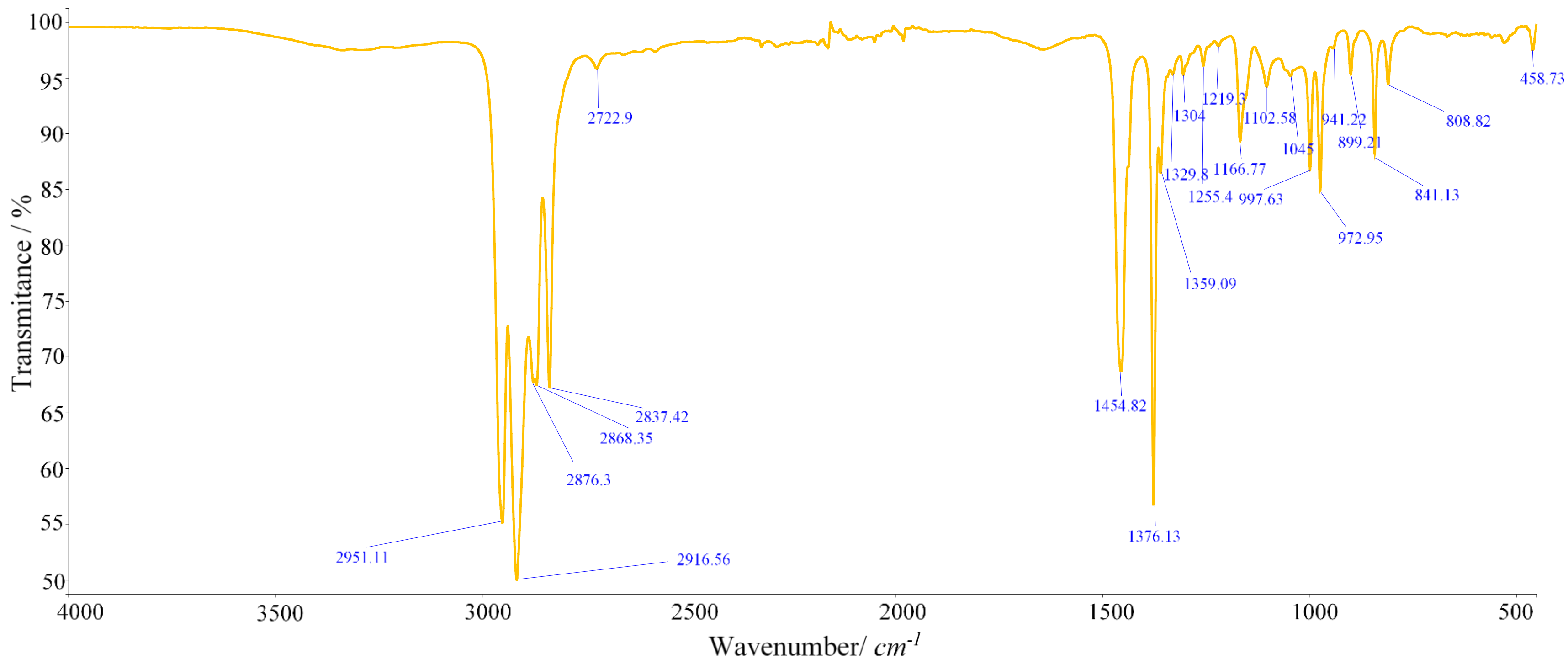

Figure 39. FTIR spectrum of recycled single-use face mask

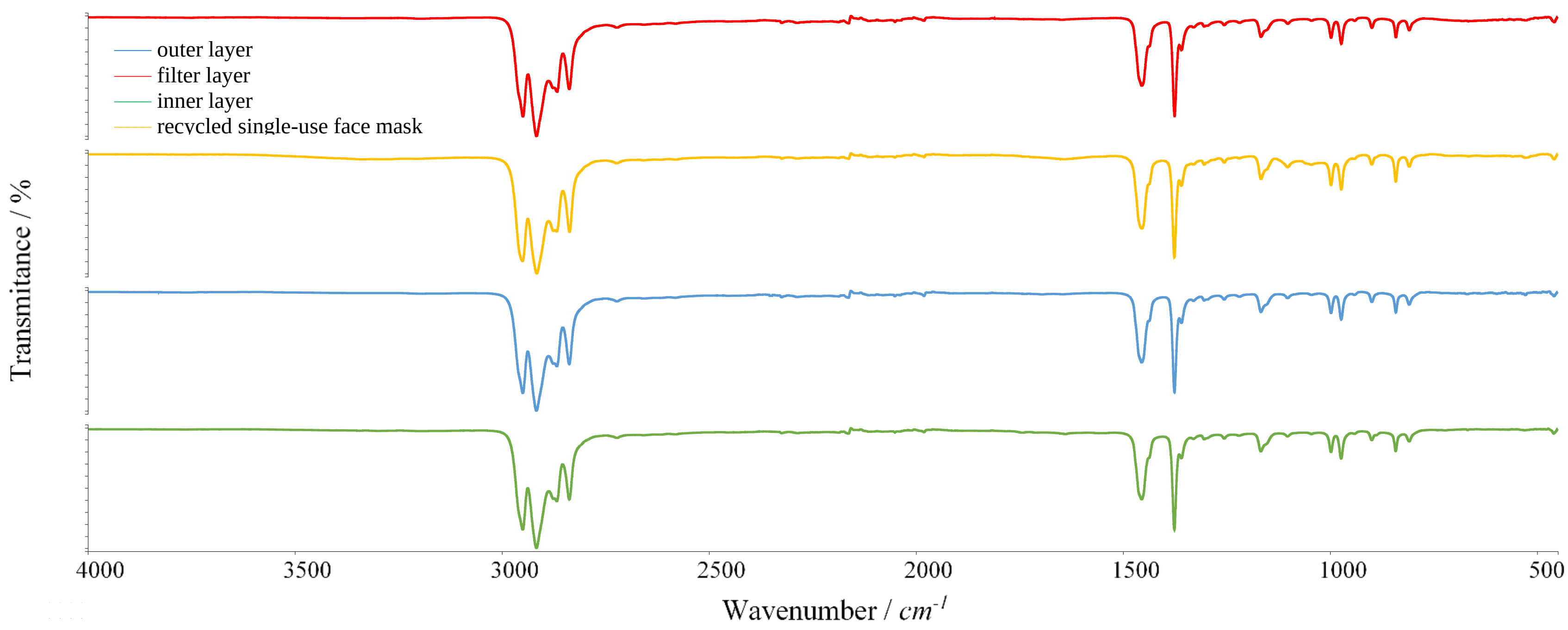

Figure 40. FTIR spectrum of outer layer, filter layer, inner layer and recycled single-use face mask

3.2.1. Differential scanning calorimetry

DSC curves of the first heating scan, cooling scan and second heating scan for single-use face mask (*Fig 41*) and recycled single-use face mask (*Fig 42*) with thermal transition parameters determined from DSC curves are shown in *Table 8*. While *Fig 43-45* shows a comparison of each thermal transition.

In comparison with single-use face mask can be concluded that mechanical recycling of single-use face mask moved T_{eim}, T_{pm}, T_{efm}, T_{eic}, T_{pc} and T_{efc} by several degrees to lower and higher temperatures, respectively. The temperature of melting in both first and second heating for mechanically recycled single-use face mask is the same, 166 ^{o}C (T_{pm}). The crystallization and melting temperatures in the recycled single-use face mask are higher (8 ^{o}C) as compared to a single-use face mask.

Single-use face mask showed two melting peaks in both first and second heating, which is related to the filter layer as described in chapter 3.1.2. While, recycled single-use face mask showed only one melting peak.

As can be seen from *Table 8* for recycled single-use face mask melting and crystallization starts and ends at higher temperatures. After recycling, the value of X_c increased. Also, the heat of melting increased up to 33 J g^{-1} (1st heating) and 17 J g^{-1} (2st heating), while heat of crystallization up to 11 J g^{-1}.

DSC analysis of the recycled single-use face mask showed only one melting peak, while the single-use face mask showed two peaks. Accordingly, it can be concluded that mechanical recycling affects the thermal stability of a single use face mask in terms of crystallinity of polypropylene, as described in chapter 3.1.2.

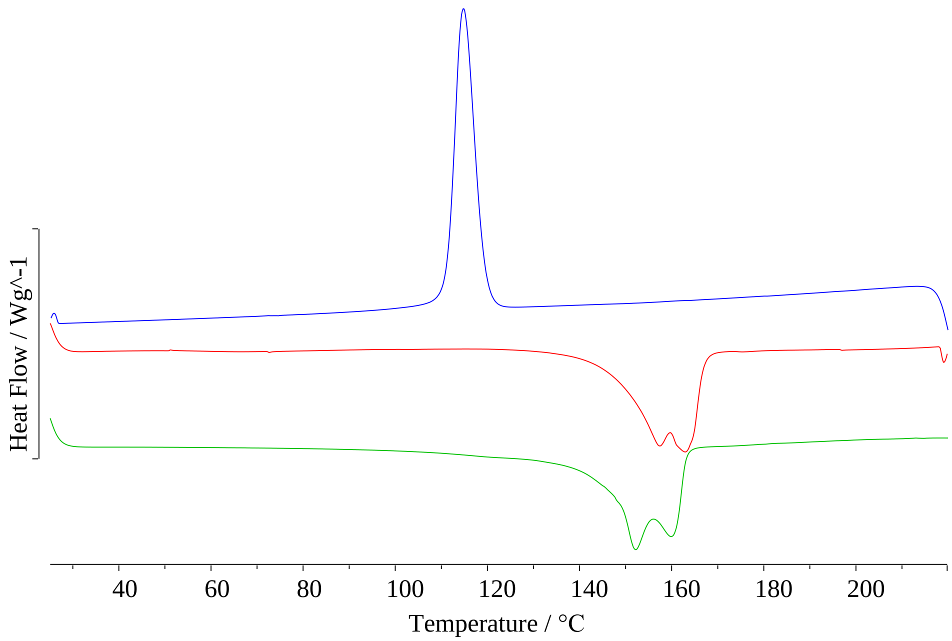

Figure 41. DSC curve of single-use face mask

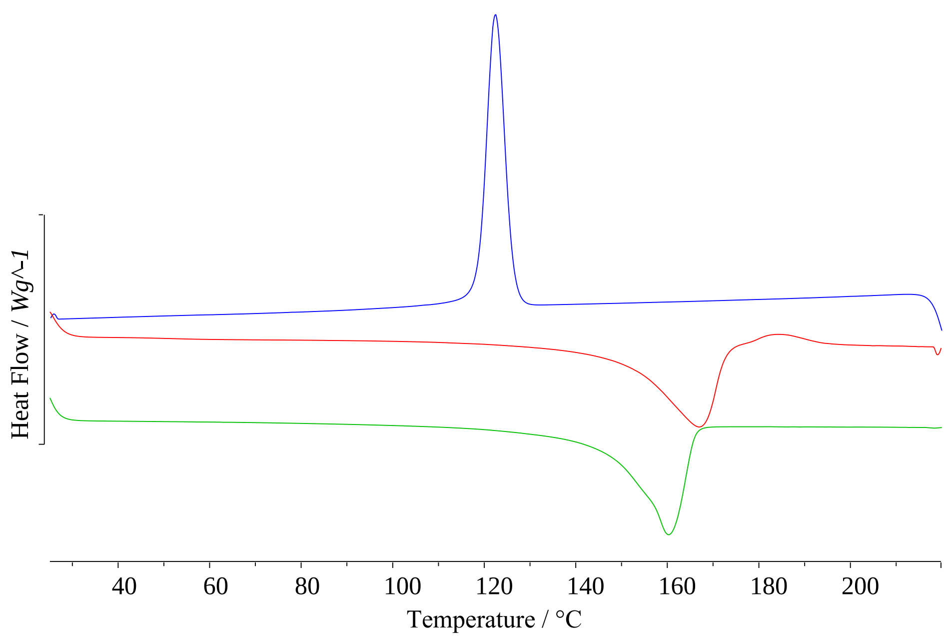

Figure 42. DSC curve of recycled single-use face mask

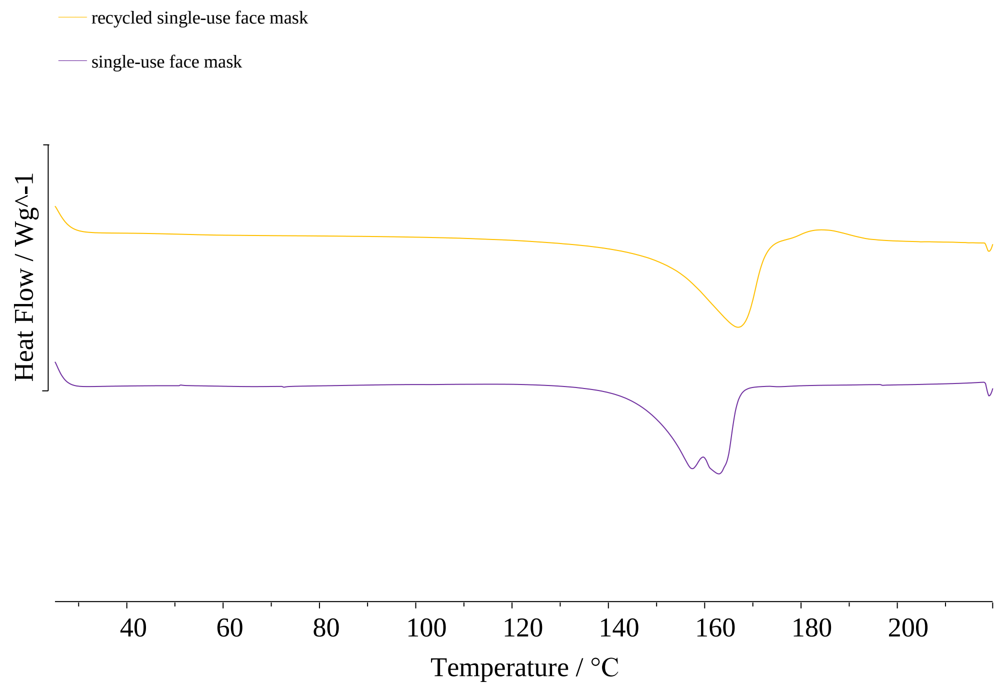

Figure 43. DSC curves of the first heating scan for single-use face mask before and after recycling

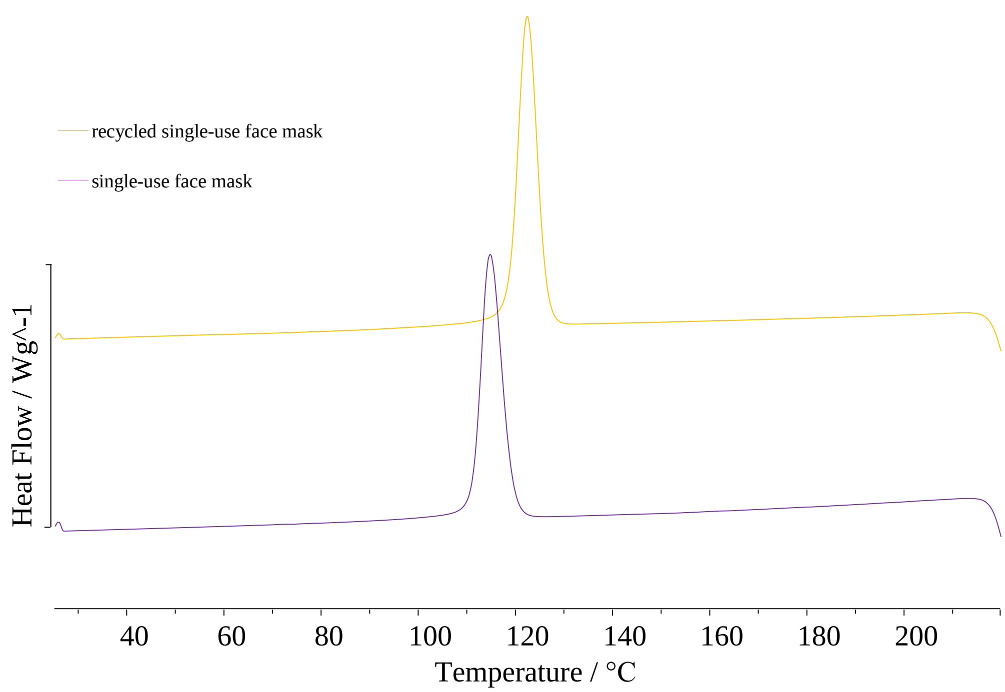

Figure 44. DSC curves of the cooling scan for single-use face mask before and after recycling

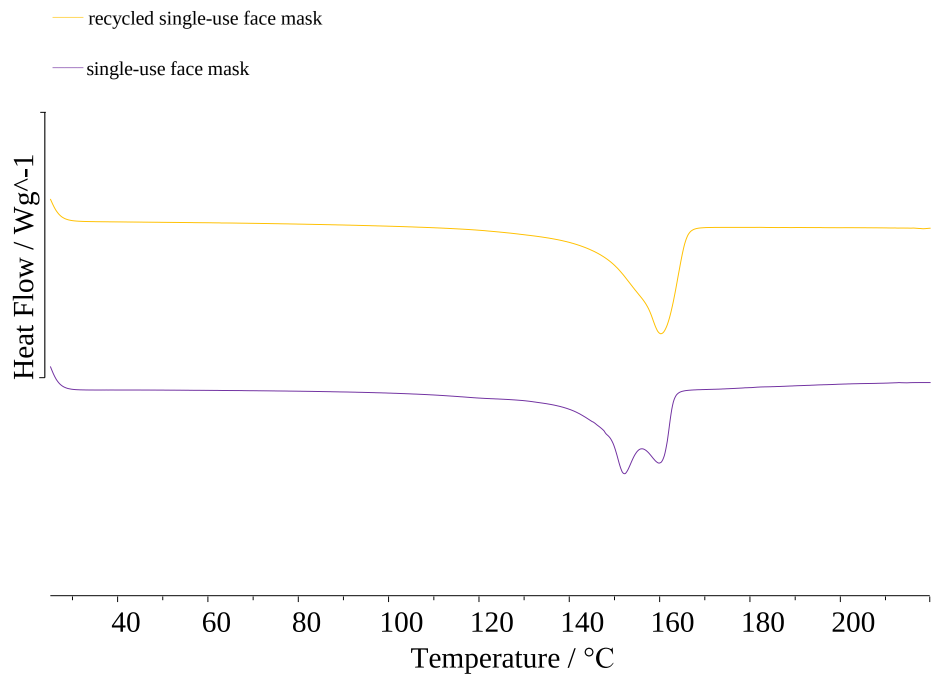

Figure 45. DSC curves of the second heating scan for single-use face mask before and after recycling

Table 8. Thermal transition parameters of single-use face mask before and after recycling

Sample	1^{st} Heating				Cooling				2^{nd} Heating				
	T_{eim} / °C	T_{pm} / °C	T_{efm} / °C	$-\Delta H_m$ / J g^{-1}	T_{eic} / °C	T_{pc} / °C	T_{efc} / °C	$-\Delta H_c$ / J g^{-1}	T_{eim} / °C	T_{pm} / °C	T_{efm} / °C	$-\Delta H_m$ / J g^{-1}	X_c / %
Single-use face mask	153	163 158	167	73.47	120	116	116	84.39	147	160 152	163	80.15	38.72
Recycled single-use face mask	148	166	173	106.32	127	124	119	95.30	151	160	166	97.36	47.03

3.2.1. Thermogravimetric analysis

Fig 46-47 shows TG and DTG curve of a single-use face mask before and after recycling. On the other hand, *Fig 48* shows a comparison of each thermal transition. *Table 9* shows parameters determined from TG and DTG curves.

As it can be seen, a single-use face mask after recycling decomposes in one stage, presenting the decomposition at an approximate temperature range 370-490 °C, while single-use face mask decomposes in two stages, where the second peak in the temperature range 310-370 °C can be correlated to the filter layer.

From the obtained results shown in *Table 9*, it can be concluded that recycling does not affect the thermal stability of a single-use face mask. Peak in the temperature range of 310-370 °C for single-use face mask has very low intensity and does not appear in recycled single-use face mask after extrusion. Accordingly, there is no significant difference in TG and DTG curves of single-use face mask before and face mask after recycling, as can be seen from *Fig 45* and *Fig 46*.

Changes in value of the onset temperature are not significant. Also, the remaining mass at the end of the thermal decomposition process is not significant, the sample decomposed almost without residue.

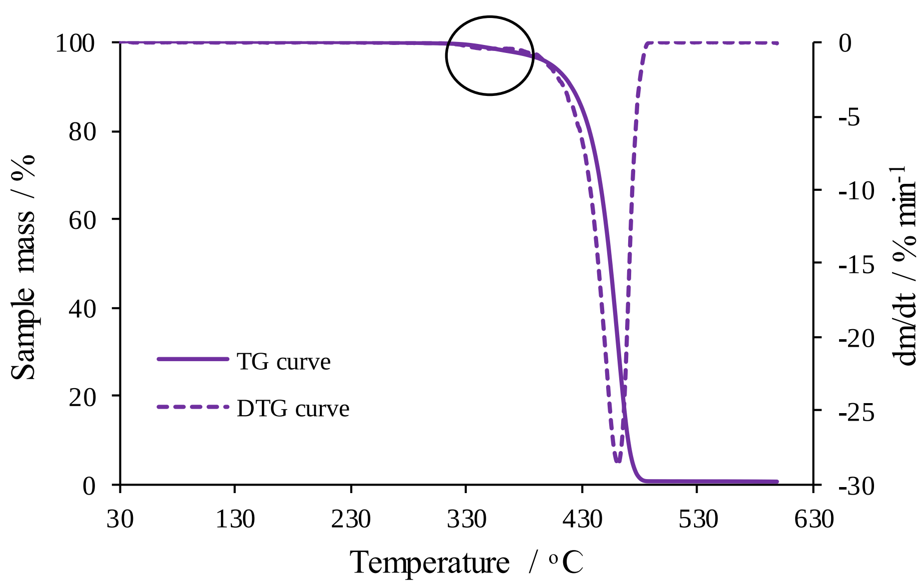

Figure 46. TG and DTG curve of single-use face mask

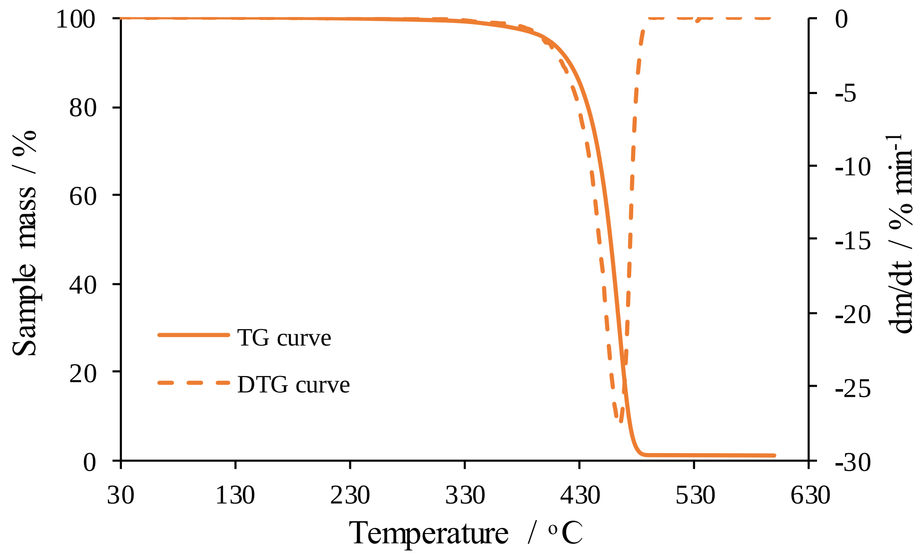

Figure 47. TG and DTG curve of recycled single-use face mask

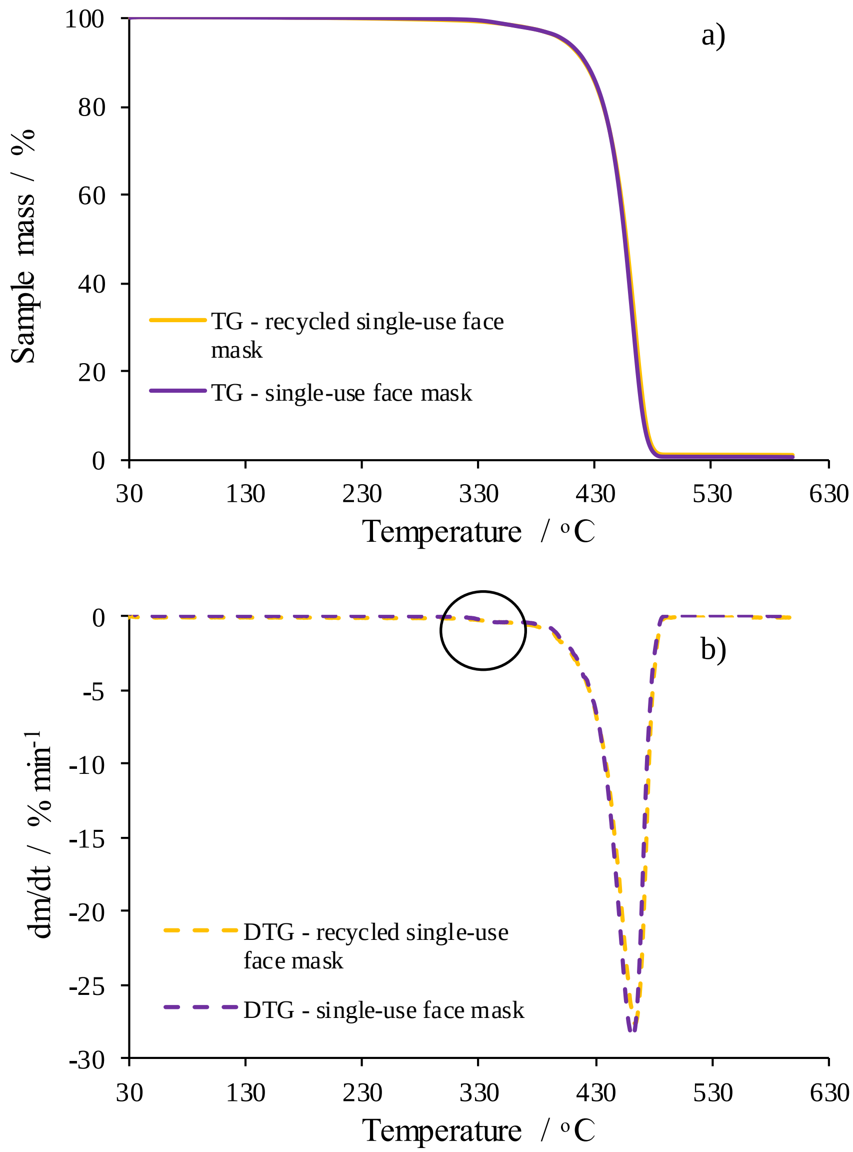

Figure 48. TG (a)) and DTG (b)) curves of single-use face mask before and after recycling

Table 9. TG and DTG characteristics of single-use face mask before and after recycling

Sample	$T_{5\%}$ / °C	T_{onset} / °C	T_{max} / °C	R_{max} / % min^{-1}	Δm / %	m_f / %
Single-use face mask	403	319	350	0.4	2.2	97.8
	/	438	462	28.7	97.1	0.7
Recycled single-use face mask	402	437	464	27.7	99.1	0.9

4. CONCLUSION

The aim of this work was to investigate the possibility of mechanical recycling of single-use face masks. FTIR, TG and DSC were used in order to determine the structure and thermal properties of each layer of single-use face mask, single-use face mask before and after recycling. As well, to identify the main polymer from which single-use mask is made of. Based on the obtained results, it can be concluded:

- FTIR analysis confirmed that the main polymer that composes each layer of a single-use face mask is polypropylene.
- Double melting endotherms for filter layer and single-use face mask have been attributed to transitions between different modifications of α crystal form, where the lower temperature endothermic peak is related to the melting of the crystals formed during the non-isothermal crystallization and the higher temperature is caused by the melting of the crystals after the recrystallization. Mechanical recycling affects the thermal stability of a single use face mask in terms of crystallinity of polypropylene.
- No exact explanation for peak in the temperature range of 300-370 $^{\circ}$C for filter layer and single-use face mask can be provided only by TG analysis.
- Mechanical recycling of single-use face masks has potential for additional processing and reuse of single-use face masks for industrial exploitation, but need more research for future development.

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