Mechanical Recycling of Disposable Protective Masks

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Abstract

Disposable protective face masks, the most used personal protective tools during the Covid-19 pandemic, have become a serious environmental concern that needs urgent attention. In this study, disposable protective face masks were disassembled into their components and mechanically recycled through extrusion. Fourier transform infrared spectroscopy and scanning electron microscopy were employed to investigate the effect of recycling on the structure and morphology of the mask material. Microscopy revealed morphological differences among the layers of the mask. Differential scanning calorimetry and thermogravimetric analysis were used to characterise the thermal properties of the samples before and after recycling. Although the mechanical recycling process had minor effect on the thermal properties and stability of the mask material, thermal methods verified differences between the layers of the mask.

Keywords

Disposable protective mask, mechanical recycling, extrusion, polypropylene

1 Introduction

Disposable protective face masks (DPMs) are the most used personal protective equipment globally. This was especially evident during the Covid-19 pandemic where masks played a crucial role in mitigating the spread of the virus through droplets and aerosols. Unfortunately, the mismanagement of such materials, mostly polypropylene (PP), poses a potential environmental threat. Discarded DPMs often find their way into municipal waste, landfills, and possibly rivers and oceans, contributing to the growing problem of microplastic pollution. Additionally, the synthetic fibres in DPMs can contribute to microfibre pollution. This issue demands attention as disposable masks have been found to act as carriers of pollutants and release heavy metals.1 Therefore, it is necessary to rethink recycling and repurposing routes to reduce their impact on the environment. Various studies conducted between 2020 and the present have proposed methods for recycling and reusing DPMs. $^{\rm 2-14}$ The primary goal is to reduce, reuse, and recycle. Four potential recycling methods have been identified:¹³ primary recycling (re-extrusion) an easy process capable of converting plastic waste into quality products; secondary recycling where the plastic waste is transformed into a new product by mechanical recycling involving segregation, purification, shredding, chopping, and granulation; tertiary recycling as a chemical process that converts a large macromolecule into a smaller molecule by depolymerisation employing heat, chemical agents, and catalysts; and quaternary recycling where energy is recovered from the waste plastic by incineration. Battegazzore et al.3 conducted a study involving morphological and

thermal analysis of mechanically extruded surgical face masks. The authors separated the surgical mask into three components: face mask (three layers), ear loop, and nose wire. They concluded that the extruded mask material could be directly recycled through extrusion and injection moulding. The main goal of their investigation was to devise a recycling scheme that would be adequate to obtain a suitable recycled material with the desired properties. However, a key challenge of mechanical recycling is the heterogeneous composition of most masks. Less conventional recycling methods, such as incorporating shredded masks into concrete aggregate for road base applications or transforming PP material into cathodes for supercapacitors, have also been explored.⁵ Recycled face masks have also been used in the production of a sustainable cement mortar.¹⁴ Regardless of the chosen recycling method, Torres et al.⁴ emphasised the need to disinfect the used masks before introducing them into the recycling stream, which would definitely increase reprocessing costs. The financial advantages of recycling plastics are significant and cannot be disregarded. However, they are often overlooked since they depend on energy savings. Nevertheless, recycling plastic waste, instead of using virgin materials, can reduce production costs, energy expenses, and negative impacts on the environment.¹³ Hence, future studies of this kind should correlate with appropriate economic analyses, especially during the industrial scale-up of the processes.

In this study, DPMs were purchased and manually disassembled into their components. The obtained filter fabric material was separated into three individual layers, manually shredded into smaller pieces, and subjected to mechanical recycling. The benefits of mechanical recycling have been highlighted in our previous work.¹⁵ The main objective of this research was to assess the effect of the mechanical recycling process, specifically extrusion, on the morphological, structural, and thermal properties of the masks' filter material.

https://doi.org/10.15255/KUI.2023.048 KUI-12/2024 Original scientific paper Received October 13, 2023 Accepted December 14, 2023

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4th ZORH CONVENTION

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Note: The findings of this study were presented at the 4th International Convention of Scientists, Specialist Employees, and Students on the topic of Environmental Protection in the Republic of Croatia (4th ZORH Convention), held on April 20–21, 2023, at the Faculty of Chemistry and Technology University of Split, Croatia.

2 Experimental

2.1 Materials

The DPMs utilised in this study were purchased from the general importer Techno Mag (Croatia), sourced from the manufacturer Guangdog Annuochen Medical Supplies Co. Ltd. (China). According to the manufacturer, the masks were a 3-ply ear-loop type, composed of 67 % non-woven and 33 % meltblown materials. The masks' dimensions were 17.5×9.5 cm, designed for the adult age group. The material type was not specified on the packaging; only the executive standard GB/T32610-2016 was indicated.



- *Fig.* 1 Disposable protective mask disassembled into its components
- Slika 1 Jednokratna zaštitna maska odvojena na sastavne dijelove

2.2 Material recycling

Before mechanical recycling, components of the disposable protective mask, namely nose wire (NW, metal material), ear loops (EL, elastomer material), and fabric material (FM), were manually separated (Fig. 1). Subsequently, the fabric material was separated into layers: inner layer (IL), filter layer (FL), and outer layer (OL) (Fig. 2). At this point, it is crucial to note that only the fabric material underwent mechanical recycling. While all three layers were extruded together, the separation allowed for the analysis of individual layers to assess the effect of extrusion on the thermal properties of the recycled material. Recycling was performed via a hot melt extrusion process in a laboratory single-screw extruder at a temperature of 160 °C with a screw speed of 100 rpm. To facilitate the extrusion process, the fabric material was manually cut into smaller pieces with scissors.



- *Fig. 2* Three individual layers separated from the fabric material of the mask
- Slika 2 Jednokratna zaštitna maska odvojena na sastavne dijelove

2.3 Material characterisation

To gather information, each individual layer separated from the fabric material, as well as the extruded sample obtained after the mechanical recycling process, were characterised using various techniques.

2.3.1 Fourier transform infrared spectroscopy

Fourier transform infrared spectroscopy (FT-IR) spectra of the investigated samples were collected using a Perkin Elmer Spectrum Two FT-IR spectrometer. The universal attenuated total reflectance (UATR) technique with a diamond reflection crystal was employed. The spectra were recorded at room temperature in 10 scans at a resolution of 4 cm⁻¹ within the range of 4000–450 cm⁻¹.

2.3.2 Differential scanning calorimetry

The thermal characteristics of the investigated samples, before and after extrusion, were determined using a Mettler Toledo DSC823e calorimeter in a nitrogen atmosphere (30 cm³ min⁻¹). The samples (10 ± 0.5 mg) were pressed in aluminium pans of 40 µl, heated (10 °C min⁻¹) from 25 to 220 °C, cooled at the same rate to 25 °C, and reheated to 220 °C. The melting temperatures (T_m), crystallisation temperatures (T_c), and corresponding enthalpy of melting and crystallisation ($\Delta H_m/\Delta H_c$) were determined according to the ISO 11357-3 international standard.¹⁶ More information can be found in our previous work.¹⁵ The degree of crystallinity was calculated according to Eq. (1).

$$X_{\rm c} = \frac{\Delta H_{\rm m}}{\Delta H_{\rm m}^0} \tag{1}$$

 ΔH_m and ΔH_m^0 are apparent melting enthalpies per gram of mask material and of 100 % crystalline PP (208 J g⁻¹),¹⁷ respectively.

2.3.3 Scanning electron microscopy

Surface morphology analysis of the separated layers of the masks' fabric material was conducted using a field emission scanning electron microscope (FEG SEM) Thermo Scientific Quattro S (FEG SEM, Hillsboro, OR, USA). The samples were coated with carbon and examined under high vacuum at 10 kV.

2.3.4 Thermogravimetric analysis

The thermal stability of the investigated samples was assessed using a PerkinElmer TGA 8000 within the temperature range of 30–600 °C. The samples (10 ± 0.5 mg) were thermally degraded in a nitrogen atmosphere (40 cm³ min⁻¹) at a heating rate of 10 °C min⁻¹. The degradation process yielded thermogravimetric curves (TG) and corresponding derivative thermogravimetric curves (DTG). The following characteristics were determined for each degradation step: onset temperature (T_{onset}), temperature



Fig. 3 – Comparison of the FT-IR spectra of IL, FL, OL, and RDPM samples *Slika* 3 – Usporedba FT-IR spektara IL, FL, OL i RDPM uzoraka

at 5 % mass loss ($T_{5\%}$), temperature at the maximum degradation rate (T_{max}), maximum degradation rate (R_{max}), final mass (m_i), and mass loss (Δm).

3 Results and Discussion

3.1 Fourier-transform infrared spectroscopy

As the exact material composition of the DPM fabric part was not specified by the manufacturer, the FT-IR spectra of the IL, FL, OL, and recycled disposable protective mask sample (RDPM) were individually collected, and are presented in Fig. 3.

Utilising the Perkin Elmer Spectral Libraries integrated into Spectrum software, all investigated samples were subjected to a *Search and Comparison* process. The results revealed that PP was indeed the material composing all components of the DPM fabric material. The correlation factor (database spectra) for all the samples was 0.995 (max. 1.000). Typical absorptions bands of PP reported in the literature² are highlighted in Fig. 3. No discernible differences in peak position (wave number), shape, and intensity between the RDPM and the three layers of the fabric material were observed, indicating that the extrusion conditions were appropriate, and that the mechanical recycling process had not affected the fabric material's structure.

3.2 Differential scanning calorimetry

Differential scanning calorimetry (DSC) was employed in three ways. Firstly, DSC served as a verification tool following FT-IR analysis, confirming that PP was the primary material of the all DPM fabric components. Normalised DSC curves for all three components of the mask are presented in Fig. 4.

All samples exhibited specific melting transitions in the temperature range of 150-170 °C with the peak temperature at around 160 °C (Fig. 4(a,c)), corresponding to the

melting temperature of PP¹⁷ (Table 1). This information guided the determination of correct recycling parameters, representing the second application of DSC. The value of 160 °C was set as the extrusion temperature of the DPM. Finally, the third application of DSC was to determine if the mechanical recycling process, specifically hot melt extrusion, affected the thermal properties of PP, the primary material of the mask. Fig. 4 shows the normalised DSC curves of the first and second heating cycles (a, c), along with the cooling curve (b). The first heating cycle aimed to eliminate the thermo-mechanical stress remaining after production and extrusion.

It is evident that all four samples exhibited a specific endothermic transition characteristic of PP, Fig. 4(c). However, the filter layer sample displayed two clearly separated peaks at 152 and 160 °C. This sample also exhibited the lowest enthalpy of melting and crystallisation, and consequently, the lowest degree of crystallinity (Table 1). This difference can be explained by the structural nature of PP, which can exist in several crystal modifications depending on crystallisation conditions and isotacticity. The monoclinic α -modification is predominant in a structure formed during slow cooling from the melt.18 The source of the double-peak transition could be related to α crystals. Specifically, double melting endotherms have been attributed to transitions between different modifications of the 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. Researchers have also proposed that a 'continuum' of different structures from the limiting disordered modification α_1 to the limiting ordered modification α_2 could exist instead of only two limiting forms.¹⁹ For α -PP, the lower temperature endothermic peak corresponds to the melting of the crystals formed during non-isothermal crystallisation, whereas the higher temperature is attributed to the melting of the crystals after recrystallisation/reorganisation, characterised by higher stability and perfection.²⁰ If we also consider the data from the cooling phase, it becomes evident that, despite the presence of PP indicated by melting, the crystallisation from the melt varies among the three lay-



Fig. 4 – Comparison of the normalised DSC curves of the investigated samples: (a) first heating, (b) cooling, and (c) second heating *Slika* 4 – Usporedba normaliziranih DSC krivulja istraživanih uzoraka: (a) prvo zagrijavanje, (b) hlađenje i (c) drugo zagrijavanje

Table 1	– Thermal	transition p	parameters	of the	investigated	samples
Tablica 1	– Značajk	e toplinskih	prijelaza is	straživa	inih uzoraka	

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Sample	1st Heating 1. zagrijavanje			Cooling hlađenje				2 nd Heating 2. zagrijavanje					
Uzorak	T _{eim} ∕°C	T _{pm} ∕°C	T _{efm} ∕°C	$-\Delta H_{\rm m}/{\rm Jg^{-1}}$	T _{eic} ∕°C	T _{pc} ∕°C	T _{efc} ∕°C	$-\Delta H_{\rm c}/{\rm Jg^{-1}}$	T _{eim} ∕°C	T _{pm} ∕°C	T _{efm} ∕°C	$-\Delta H_{\rm m}/J{\rm g}^{-1}$	X _c /%
IL	159	163	170	88.4	120	116	112	92.5	156	161	169	93.9	45
FL	148	160	163	71.0	119	114	111	77.4	147	152/161	163	80.2	39
OL	154	163	167	82.9	130	127	124	89.9	158	163	167	89.9	43
RDPM	148	166	173	106.3	127	124	119	95.3	151	160	166	97.4	47

ers, as depicted in Fig. 4(b) and Table 1. While there is no difference between IL and FL, the OL starts to crystallise 10 °C earlier. This observation suggests the presence of small amounts of other components in the FM layers, which remain undetectable by FT-IR due to the overlap with PP adsorption bands. For example, the OL layer is coloured blue, and this pigment may initiate crystallisation of PP from the melt at an earlier stage. However, it is essential to emphasize that identifying components of the FM layers is not the primary goal of this work; rather, the goal is to detect the material's thermal behaviour during extrusion. Finally, given that the RDPM sample comprised three layers, and considering the thermal transition parameters summarised in Table 1, it can be concluded that the mechanical recycling process had no discernible effect on the thermal properties of the fabric material. Furthermore,

the DSC curve of RDPM represents the cumulative curve of individual FM layers, as substantiated by their thermal characteristics in Table 1. This conclusion aligns with the findings of the FT-IR analysis. To provide further insights into the thermal properties of the investigated samples, TG analysis was employed.

3.3 Scanning electron microscopy

The morphological features of the three separated layers of the disposable protective mask are shown in Fig. 5 ($100 \times$ magnification). The primary purpose of the microscopic analysis was to elucidate the data obtained from FT-IR and DSC analysis. The inner and outer layers exhibited a specific non-woven fabric morphology³ and were made of uniform circular fibres (Fig. 5(c,d)). In both cases, these fibres had evidently melted together, observed as square-like melting junctions in Fig. 5(c,d).



- Fig. 5 SEM micrographs of (a) FL, (b) FL (500 \times magnification), (c) OL, and (d) IL samples
- Slika 5 SEM mikrografije uzoraka (a) FL, (b) FL (uvećanje 500×), (c) OL i (d) IL

However, the filter layer (Fig. 5(a)) can be distinguished from the other two layers by its different structure in terms of arrangement and dimensions of the fibres. As seen in Fig. 5(b), the diameter of the fibres varies significantly. Finally, SEM analysis confirmed that the difference noticed by DSC analysis originated from the fibre itself, specifically, the composition of the fibres.

3.4 Thermogravimetric analysis

A comparison of the thermogravimetric curves of the three separated DPM layers is provided in Fig. 6, while the TG/ DTG parameters are presented in Table 2.



Fig. 6 – Comparison of (a) TG, and (b) DTG curves of the investigated samples

Slika 6 – Usporedba (a) TG i (b) DTG krivulja istraživanih uzoraka

PP is a polymer that thermally decomposes through only one degradation stage in a nitrogen atmosphere.²¹ Thus, it

Table 2 – Characteristics of thermal degradation curves of the all investigated samples *Tablica 2* – Karakteristike krivulja toplinske razgradnje svih istraživanih uzoraka

Sample Uzorak	<i>T</i> _{5%} ∕°C	T _{onset} ∕°C	T _{max} ∕°C	$R_{\rm max}/\%{\rm min^{-1}}$	$\Delta m / \% \min^{-1}$	m _f /%
IL	397	435	464	23.9	98.4	1.6
FL	365	315/434	341/465	0.9/21.8	4.3/94.4	95.7/1.3
OL	407	433	462	23.8	98.9	1.1
RDPM	402	437	464	27.7	99.1	0.9

is expected that all three layers degrade through only one stage. However, according to the DSC analysis, the filter layer proves to be an exception. Fig. 6 clearly shows that the green TG/DTG curve, representing the filter layer sample, is notably different from the other two layers. It can thus be concluded that thermal degradation of the filter layer sample initiated at a lower temperature (315 °C) in comparison to the other samples (Table 2), and proceeded through two degradation stages, as graphically depicted by the two DTG peaks in Fig. 6(b). This observed exception and the variation in TG data align with the conclusions drawn from the DSC analysis. The main degradation stage of PP dominates the investigated temperature region and may overlap with some smaller degradation stages of other components present in the FM layers. Indeed, these additional components can readily be decomposed, becoming visible parallel to the PP degradation stage. Once again, the TG curve of the RDPM represents the cumulative degradation curve of all three layers in the FM.

However, the primary objective of the TG analysis was to assess the effect of mechanical recycling on the degradation pattern and the thermal stability of the recycled disposable protective mask. The RDPM sample degraded through only one degradation stage, starting at 437 °C (T_{onset}) with a T_{max} at 464 °C, leaving almost no residue (0.9 %). When compared to each separated layer of the fabric material of the mask, there is no significant difference in characteristic values, except for two DTG peaks in the FL. Therefore, from Table 2, it can be postulated that the mechanical recycling process had no discernible effect on the thermal stability of the fabric material. This serves as conclusive evidence that the extrusion conditions (160 °C and 100 rpm) for the mechanical recycling process of the disposable protective masks were appropriately selected.

4 Conclusion

The primary objective of this study was to assess the effect of the mechanical recycling process, specifically through extrusion, on the morphological, structural, and thermal properties of the filter material of disposable protective face masks. IR spectroscopy revealed no differences in spectra between the three layers of the mask and the recycled sample. Microscopy revealed differences in the structure of the three layers concerning arrangement and dimensions of the fibres. DSC and TG analyses distinguished the filter layer of the mask from the other layers, indicating differences in the enthalpy of melting and crystallisation, as well as the thermal degradation pattern. However, the mechanical recycling process had no discernible effect on the thermal stability of the fabric material of the mask, suggesting that the extrusion conditions, in terms of temperature and screw speed, were appropriately selected.

ACKNOWLEDGEMENTS

The authors would like to thank to Prof. Aleš Nagode, DSc., and Assistant Matija Zorc, DSc., from the University of Ljubljana, Faculty of Natural Sciences and Engineering, for their invaluable help with the SEM research.

List of abbreviations and symbols Popis kratica i simbola

IL	– inner layer – unutarnji sloj
FL	– filter layer – filtarski sloj
OL	– outer layer – vanjski sloj
DPM	– disposable protective face mask – jednokratna zaštitna maska
PP	– polypropylene – polipropilen
RDPM	– recycled disposable protective face mask – reciklirana jednokratna zaštitna maska
FTIR	 Fourier transform infrared spectroscopy infracrvena spektroskopija s Fourierovom transformacijom
UATR	– universal attenuated total reflectance – univerzalna totalna prigušena refleksija
DSC	– differential scanning calorimetry – diferencijalna pretražna kalorimetrija
TG	– thermogravimetric analysis – termogravimetrijska analiza
SEM	– scanning electron microscopy – pretražna elektronska mikroskopija
T _m	– melting temperature – temperatura taljenja
$T_{\rm c}$	– crystallisation temperature – temperatura kristalizacije
$T_{\rm eix}$	– extrapolated onset temperature – ekstrapolirana početna temperatura (x = m, c)
$T_{\rm px}$	– peak temperature – temperatura u minimumu prijelaza (x = c, m)
$T_{\rm efx}$	– extrapolated end temperature – ekstrapolirana konačna temperatura (x = m, c)
$\Delta H_{\rm m}$	– enthalpy of melting – toplina taljenja
$\Delta H_{\rm c}$	– enthalpy of crystallisation – toplina kristalizacije
X _c	– degree of crystallinity – stupanj kristalnosti
$T_{\rm onset}$	– onset temperature – temperatura početka razgradnje
T _{5%}	 temperature at 5 % mass loss temperatura pri kojoj uzorak izgubi 5 % početne mase
T _{max}	 temperature at the maximum degradation rate temperatura pri maksimalnoj brzini razgradnje
R _{max}	– maximum degradation rate – maksimalna brzina razgradnje
m _f	– final mass – konačna ostatna masa
Δm	– mass loss – gubitak mase za pojedini stupanj razgradnje

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SAŽETAK

Mehaničko recikliranje jednokratnih zaštitnih maski

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Jednokratne zaštitne maske (DPM), kao najčešće upotrebljavani alat tijekom SARS-Covid-19 pandemije, postale su ozbiljan ekološki problem koji je potrebno riješiti. U ovom radu jednokratne zaštitne maske razdvojene su na sastavne komponente i mehanički reciklirane ekstrudiranjem. Infracrvena spektroskopija s Fourierovom transformacijom i pretražna elektronska mikroskopija primijenjene su za određivanje utjecaja recikliranja na strukturu i morfologiju materijala maske. Mikroskopija je ukazala na postojanje morfoloških razlika između slojeva maske. Toplinska svojstva istraživanih uzoraka prije i poslije recikliranja karakterizirane su primjenom diferencijalne pretražne kalorimetrije i termogravimetrijske analize. Iako su toplinske metode analize potvrdile razliku između slojeva maske, može se zaključiti da mehaničko recikliranje nije znatno utjecalo na toplinska svojstva i toplinsku stabilnost materijala maske.

Ključne riječi

Jednokratna zaštitna maska, mehaničko recikliranje, ekstruzija, polipropilen

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Izvorni znanstveni rad Prispjelo 13. listopada 2023. Prihvaćeno 14. prosinca 2023.