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Original Research Article

PP Smoothing Process Using Green Solvents

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Abstract: Novel surface treatment for Additively Manufactured Polypropylene parts is presented in this work for potential uses in medical applications. The method uses green sustainable solvent to smooth, seal and remove semi-sintered powder particles from the surface. The new surface smoothing with given solvent produces parts that can be used in medical applications. Optical microscope images of the smoothed part showed disappearance of the semi-sintered powder particles and surface pores, which were largely visible on the unprocessed part. The smoothed PP parts were seen to have better performance properties, both mechanically and aesthetically. Cytotoxicity results showed that the smoothed PP parts have no cytotoxic effect.

I. Introduction

Polypropylene (PP) is a non-polar, semi-crystalline thermoplastic polymer belonging to the polyolefin group. PP is a commonly used thermoplastic, and it is very cost effective to purchase as well. The properties of PP are dependent on its crystallinity, molecular weight, and stereochemistry (tacticity). PP has excellent chemical-resistant and water-impermeability properties. The recyclability of PP alongside its properties makes it suitable for many industrial applications [1]. PP finds a lot of relevance in diverse aspects of medicine. It is used in the manufacture of medical products such as syringes, pouches, hospital disposables, test tubes, beakers, and pipettes. Drug-delivery systems, packaging and non-woven fabrics used in hospitals are made from PP. The water-impermeability property of PP makes it an attractive choice in the manufacture of biocompatible implants [2]. One of the main hindrances to the use of 3D-printed PP parts in medical applications is due to the imperfections seen on the printed parts. Solvent smoothing of PP parts is very challenging; owning to the fact that PP is highly resistant to chemicals. However, identified solvents (such as carbon tetrachloride, benzene, xylene, etc.) used to smooth/dissolve PP parts have been identified as carcinogenic, neurotoxic, and environmentally unfriendly [3]. To this end, Additive Manufacturing Technologies Ltd (AMT) using its flagship 3D polymer post-processing equipment and expertise, have successfully demonstrated the smoothing of PP parts using a proprietary green solvent [4, 5].

II. Materials and methods

FA9202 (our Unique Product ID for the solvent) is the green solvent used for the post-processing of the 3D-printed polymer part in this study; it is a non-polar solvent and has low toxicity. Prosthetic cover (Ricoh PP S5500P) printed using selective laser sintering process (SLS), was supplied by a Ricoh 3D to AMT for chemical smoothing with the green solvent. It is a general calf prosthetic cover; PP as the 3D-printed polymer material, was selected for its excellent flexibility, impact resistance and waterproof properties combined with lightweight. Through the benefits of 3D printing, the part can be customized in size, shape, and texture to fit the patients' needs. The dimensions of the prosthetic PP part used in this study are 20 cm in length, 9 cm in width, and 0.3 cm in thickness. The prosthetic parts were processed with FA9202 and dried in the oven at 50 ^{o}C for 24 hours. During the processing of the PP parts, the green solvent had no chemical interactions with the additives (such as softeners/plasticizers, stabilizers/antioxidants etc.) used in the manufacture of the PP powder – this was evidenced as no leaching of additives from the PP parts was physically observed after the post-processing treatment. Photos of the unprocessed and processed parts were taken using a camera. Optical microscope images of the unprocessed and processed prosthetic parts were captured using Hirox KH-8700 digital microscope with MX(G)-2016Z lens. The surfaces of the parts were analyzed at a magnification of 20x.

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Cytotoxicity test was carried out on the post-processed part in accordance with ISO 10993-5 (2009), ISO 10993-1 (2018) and ISO 10993-12 (2012). The material was extracted for 24 hours at 37 °C and a partial pressure of 5% carbon dioxide in extraction medium (DMEM medium with antibiotics, without fetal calf serum [FCS]). The material surface / extraction volume ratio was 3cm^2 material per ml extraction medium. After extraction, the extraction medium was sterile filtered and supplemented with sterile FCS (concentration of FCS in extraction medium: 10 %). The FCS-supplemented extraction medium was pipetted under sterile conditions on pre-cultivated cells of the mouse fibroblastic cell line L929 and incubated for 48 hours at 37 °C and a partial pressure of 5% carbon dioxide. The extract was tested in four dilutions (90 %, 30 %, 10 % and 3.3 %). Each dilution was tested in four parallel experiments. Triton X 100 was used as a toxic control substance (concentration in the experiment: 1 % v/v). Cell culture medium was used as a non-toxic control. After the 48-hour incubation period, the protein content of the cell culture was determined by the method.

III. Results and discussion

FA9202 smoothed the prosthetic cover well and the appearance of the smoothed part was remarkable (as shown in Figure 1a).

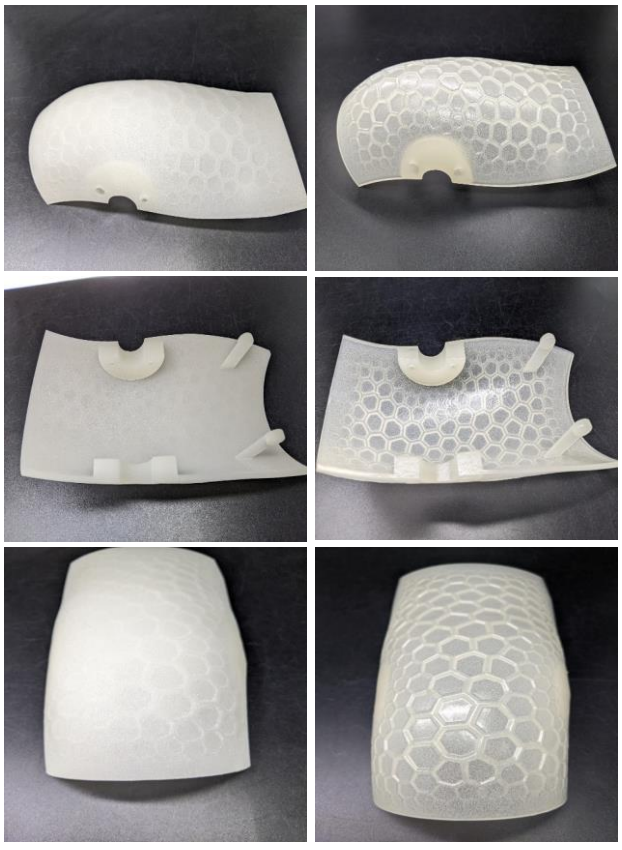


Figure 1a. Unprocessed part (left) vs Processed part (right). part used in this study are 20 cm in length, 9 cm in width, and 0.3 cm in thickness

It was observed that the part lines and impressions were more visible and apparent on the processed part after the smoothing process. Semi-sintered particles seen on surface of the unprocessed part were removed after the chemical smoothing. Considerable increase in luminous transmittance can be observed on the processed part. Sharp edges and corners of the component were maintained after the smoothing process (see Figure 1b).

Figure 1b. Sharp edges and corners of unprocessed part (left) vs processed part (right)

Optical microscope images (µm scale) revealed the presence of semi-sintered particles and surface porosity on the unprocessed part.

The surface morphology of the processed part showed that there were no traces of semi-sintered particles, and the pores have completely disappeared (see Figure 2).

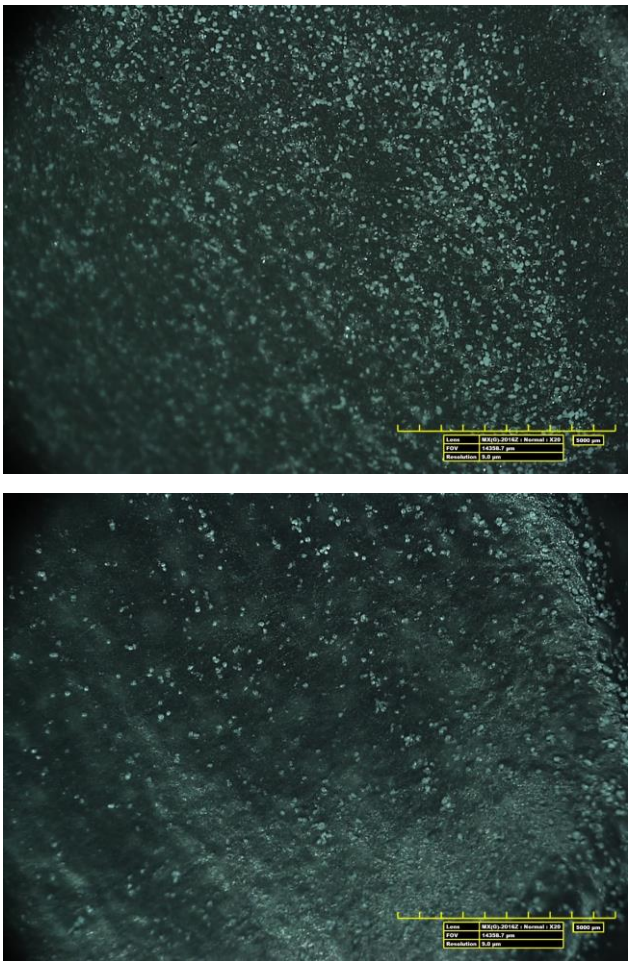


Figure 2. Optical images of (a) unprocessed part and (b) processed part. The optical microscope images were captured at 5000 µm scale.

The absence of the semi-sintered particles on the processed part was physically evidenced/observed as the level of smoothness was drastically increased. The surface roughness measurements on the unprocessed and processed parts were evidence to the presence and absence of the semi-sintered particles respectively.

The surface roughness Ra of the PP parts was measured using a Mitutoyo Surftest SJ-210 with a stylus tip radius of 2µm, tip angle 60° and measuring force 0.75kN. Five measurements at different areas of each part's surface were made before and after processing and the average calculated. The surface roughness measurement for each part is shown in Figure 3.

A conventional injection-moulded surface roughness Ra for PP can be found to be within 0.1 – 0.5µm. The average roughness Ra for unprocessed/as-printed PP part was measured to be 12.35µm, whereas the measured roughness Ra for the processed PP part was found to be within the 0.46 and 1.80µm.

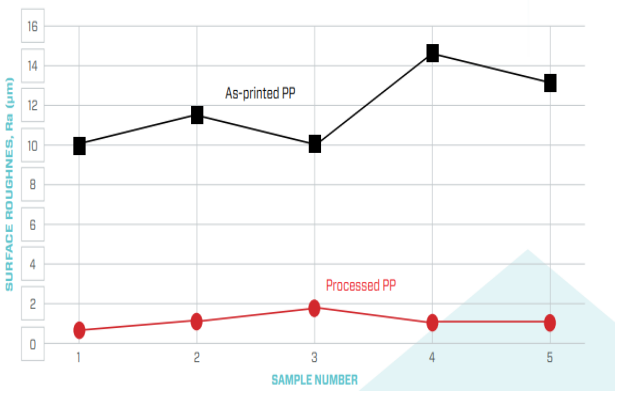


Figure 3. Surface roughness of PP parts (before and after processing) using Mitutoyo Surftest SJ-210

The mechanical properties of the unprocessed/as-printed PP parts were seen to improve after the post-processing treatment (see Figure 4).

There was a slight increase in the tensile strength of the processed parts. An improvement was also observed at the break stress with an average value of 19MPa for the processed parts versus the average value of 13MPa seen for the unprocessed parts.

The average elongation at break for unprocessed tensile parts was measured at approximately 93%, and the processed parts recorded an average elongation at break of approximately 355%.

This is an increase of more than three and a half times the elongation of the unprocessed/as-printed parts. The Young's Modulus results did not show any significant change for both the unprocessed and processed parts.

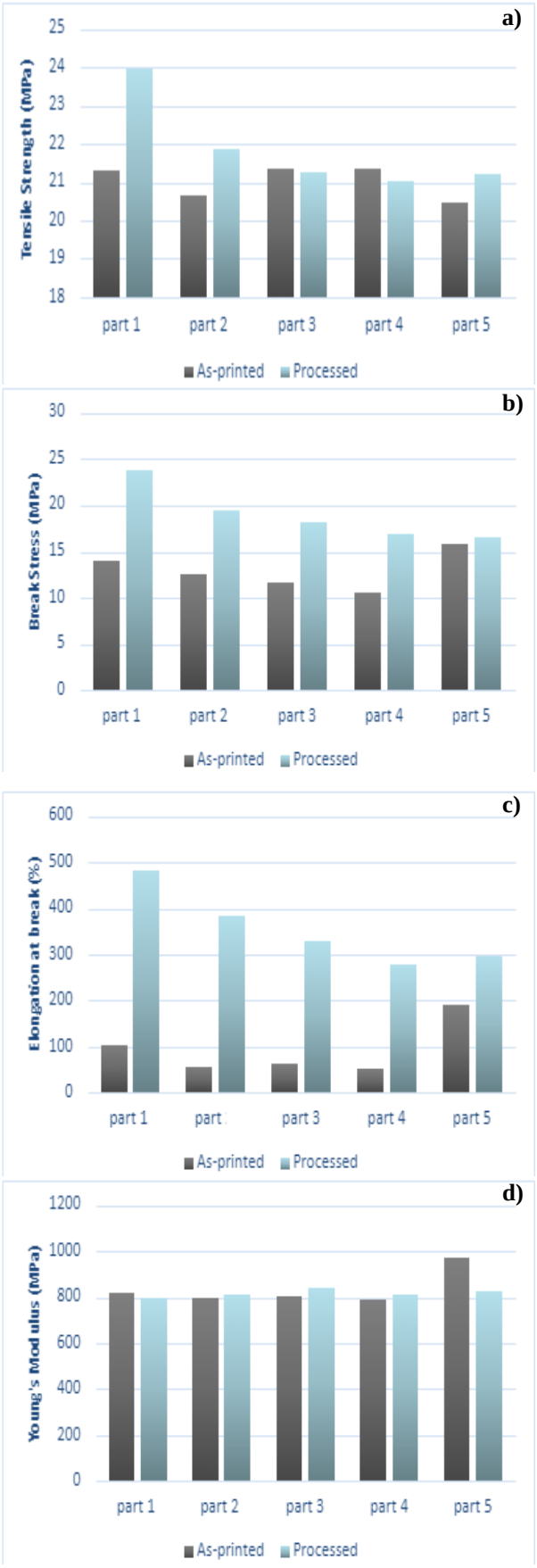
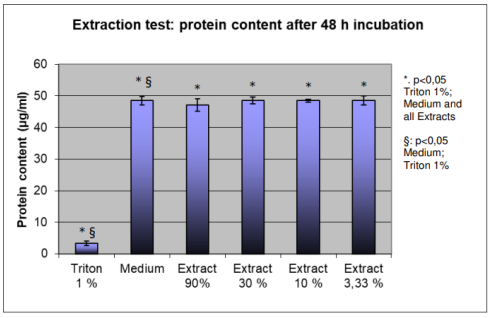


Figure 4. Mechanical properties of the unprocessed/as-printed PP parts versus the processed PP parts (a) Tensile strength results (b) Break stress results (c) Elongation at break results and (d) Young's Modulus results.

The prosthetic cover used in this paper is a scaled-down version (dimensions: 20cm long, 9cm wide and 0.3cm thick) of the prosthetic leg cover (see Figure 5). The prosthetic leg cover is designed to provide balance to the silhouette of the leg and improve the confidence of users of knee prostheses.

Figure 5. Prosthetic leg cover

The cytotoxicity result revealed that there is no cytotoxic effect on the PP part treated with FA9202 (see Figure 6). Materials are said to be cytotoxic, if the material extract leads to a protein content of the test cells of less than 70% of the µg/ml value compared to the negative control.



Result data	Protein content n=4					
µg/ml	Triton 1 %	Culture medium	Extract 90 %	Extract 30 %	Extract 10 %	Extract 3,3 %
Mean	3,30	48,49	47,12	48,58	48,50	48,52
Standarddev.	0,69	1,38	1,99	1,02	0,47	1,41

Figure 6. Cytotoxicity result of the processed PP part

IV. Conclusions

The SLS medical-grade PP parts have rough surface texture and therefore are not considered as good choices for medical applications.

Using AMT post-processing technology, the smoothing of the SLS PP parts is very comparable to PP parts manufactured using injection moulding process. The processing delivers significant improvements in surface smoothness, aesthetics, uniformity, and mechanical properties.

Our paper reports on the surface treatment/smoothing of 3D-printed PP parts manufactured via SLS by powder; however, the surface treatment/smoothing can also be carried out on PP parts manufactured via multi-jet fusion (MJF) and other Additive Manufacturing methods.

Furthermore, the use of FA9202 in the smoothing of SLS PP parts is an added advantage, when used for medical applications. FA9202 has low toxicity and is environmentally benign. The low toxicity of FA9202 will enable the PP parts to become biocompatible and suitable for medical uses.

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Author's statement

Conflict of interest. Research has been funded by Additive Manufacturing Technologies Ltd. Test samples haven been provided by Ricoh 3D. Both authors are employees of Additive Manufacturing Technologies Ltd. Informed consent has been obtained from all individuals included in this study.

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