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Postprocessing Techniques for Removal of Entrapped Resin in Additive Manufactured Parts

by John Brown and Thomas Plaisted

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14. ABSTRACT This report is on the effect of postprocessing conditions to remove entrapped, uncured resin from porous specimens fabricated by polymer additive manufacturing. The first removal method investigated the effect of using a centrifuge to remove resin by physical separation of the liquid from the surrounding solid structure. Samples were weighed before, during, and after timed intervals within the centrifuge. The second removal method involved the use of sonication in a solvent bath, where again samples were weighed before, during, and after timed intervals in the sonicator to determine the extent of resin removed from the samples.					
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1. Introduction

Additive manufacturing (AM) is being widely studied for a variety of reasons. It allows for rapid prototyping of intricate parts by automated machinery and with minimal wasted material, and thus serves a valuable means of fabrication in both laboratory and industrial settings. However, the quality of printed parts varies, depending on the chosen manufacturing technique, the complexity of the part geometry, and the quality of the equipment and material used in processing. Additionally, the postprocessing steps that entail removal of the part from the machine, cleaning, and final curing can further affect the quality of the build. This report seeks to quantify two techniques used in postprocessing to remove entrapped resin that improve the quality of the final part.

Stereolithography (SLA) is one of the original AM processes developed for polymers and offers a high degree of resolution compared with other AM techniques. A schematic representation of the SLA process is given in Fig. 1. A typical SLA machine cyclically raises and lowers a platform on which the parts are fabricated in a bath of photo-sensitive liquid resin. With each cycle, a blade is passed across the platform to create a uniform layer of resin. The resin layer is exposed to a UV laser that traces a cross section of the desired geometry, thereby selectively curing the resin into a specific pattern. The process is repeated as the platform is lowered into the resin and the next layer is cured on top of the previous layer. A support structure, shown in Fig. 2, is typically first built on the platform and serves as the foundation from which the part is built.

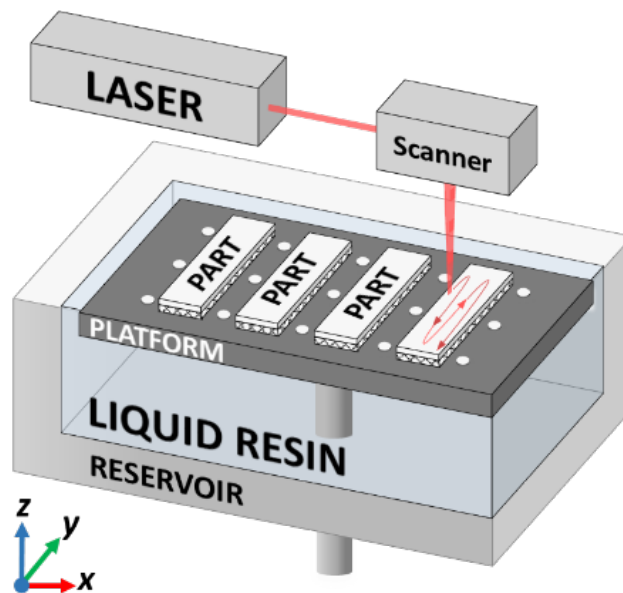


Fig. 1 SLA AM process

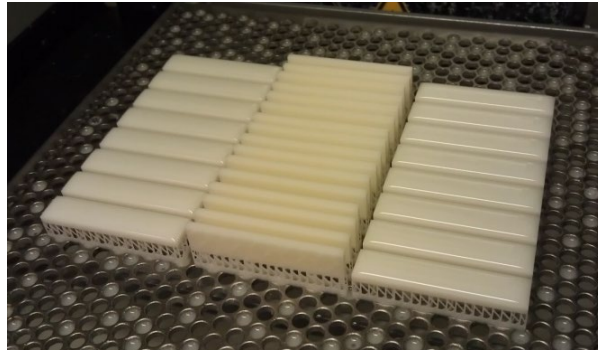


Fig. 2 Example parts fabricated by the SLA machine

Once the manufacturing process is complete, the part must be postprocessed by hand to finalize the build. This entails the following:

- Draining off excess resin that may have accumulated on the part.
- Separating the part from the build platform and removing the support structure. Typically supports are designed to be physically removed by breaking them off by hand.
- Wiping the surface of the part with a mild solvent to further remove excess resin.
- Postcuring the part with UV light or thermal treatment in an oven.

The SLA process gradually submerges the part into the resin bath as the build progresses. If the part contains fine features, such as open porous structures, liquid resin will tend to seep into these regions and can become entrapped. This trapped material can be difficult to remove, particularly if the resin has a high viscosity. If left inside the part, it will likely cure over time and become permanently bonded to the inside of the part, which may defeat the purpose of printing the fine structure in the first place.

To address this problem, we investigated two methods for removal of entrapped resin during the postprocessing step of AM. The first method, sonication, applies ultrasonic frequencies to the part to dislodge the entrapped resin from the surrounding structure. Sonic waves are transferred to the sample through a liquid medium, typically a chemical solvent that aids in thinning the entrapped resin. Sonication is often mentioned by the manufacturer of additive resins as the recommended means of removing entrapped materials. The second method, centrifugal processing, uses angular acceleration as a means of physically separating substances of different density. This technique is commonly used in

physical separation of chemical compounds, though its use in the postprocessing of additive manufactured parts has not been documented before in the open literature.

2. Experimental

2.1 Materials and Fabrication

SLA was chosen as a means to manufacture the specimen due to its ability to create high-resolution parts. The geometry of the specimens fabricated for this study were originally designed to serve as a surrogate structure for the human cranial bone (Gardner et al. 2017). As such they consist of a three-layer architecture, where the outer two layers (1.7-mm thickness) are solid, and the middle layer (2.8-mm thickness) is made up of a uniform porous structure. The porous region consists of intersecting orthogonal cylinders with characteristics as listed in Table 1. The overall sample dimensions were $60 \times 12.7 \times 6.2$ mm. A cross section of the specimen geometry is shown in Fig. 3.

Table 1 Characteristics of the pore structure for the specimens fabricated

Total solid volume (%)	Total porosity (%)	No. of pore layers	Pore diameter (μm)	Pore wall thickness (μm)
75	25	3	560	373

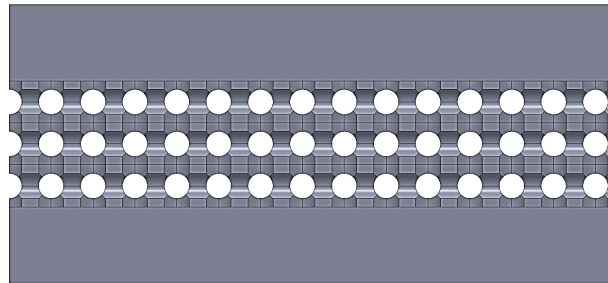


Fig. 3 Select cross-sectional view of the specimen geometry used in this study

The SLA machine used in this study to manufacture specimens was a Viper Si (3-D Systems, Rock Hill, South Carolina). An epoxy-acrylate resin with nanosilica particulate reinforcement was used to manufacture the specimens' sandwich structures. This resin system has a viscosity of around 1000 centipoise at room temperature. Ten specimens were printed for this study in a single batch. All specimens were removed from the print platform by mechanical means such as a scraper. The support structure was removed by breaking it off by hand. Then samples were individually submersed in isopropanol (IPA) solvent and agitated by

hand for 30 s and then dried with pressurized air, as shown in Fig. 4. Each specimen was then labeled and weighed individually to obtain its starting weight.

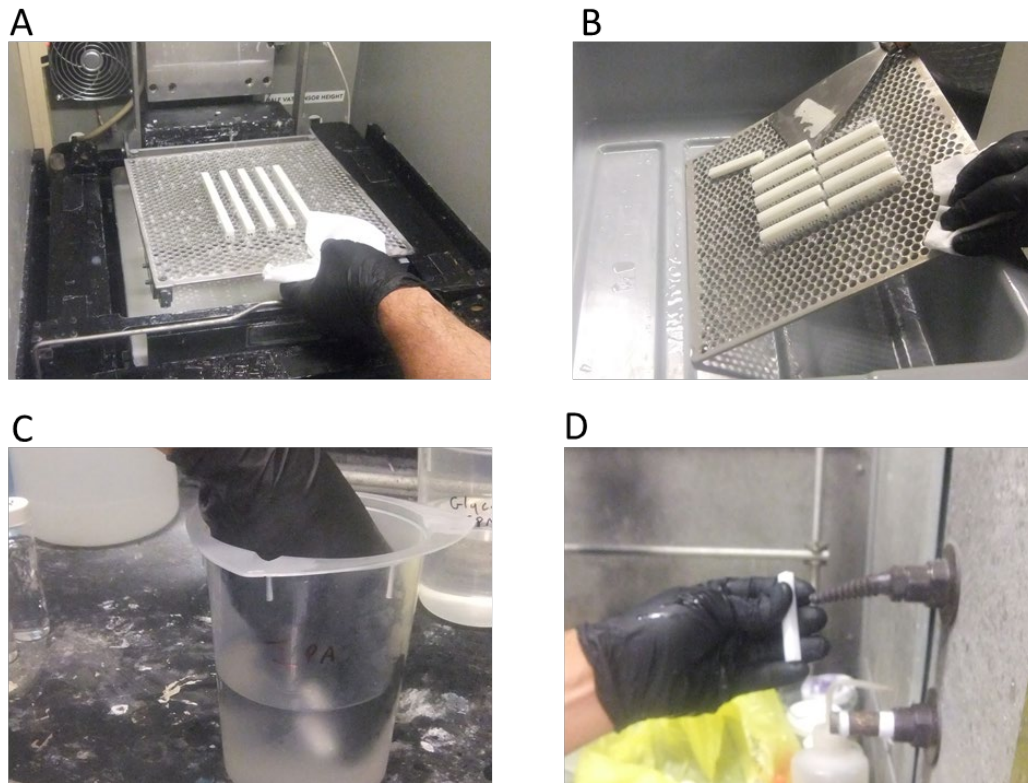


Fig. 4 Specimen fabrication by SLA: A) specimens as printed, B) removal from the build platform, C) initial rinse in IPA, and D) drying with pressurized air

2.2 Centrifuge Processing

Six of the specimens were postprocessed by spinning in a centrifuge. The centrifuge model used was an IEC Centra GP8R (Thermo Electron, Waltham, Massachusetts). The centrifuge was equipped with a bucket accessory as pictured in Fig. 5.

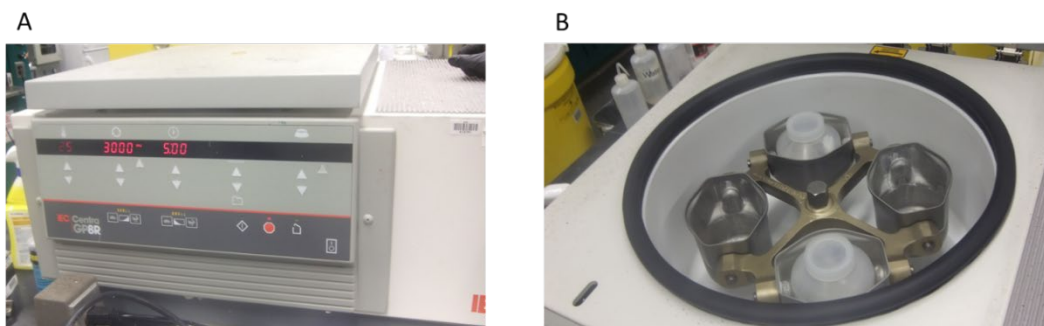


Fig. 5 Centrifuge processing: A) GPR8 centrifuge and B) sample bottles placed within the bucket holder

The centrifuge procedure was completed as follows:

- Each specimen was wrapped in small paper towel (Chemwipe).
- Three specimens were then grouped together and placed at the bottom of the centrifuge bottle and oriented such that they were lying flat on their long dimension with the pore surface facing down. Another three specimens were placed likewise within a second centrifuge bottle.
- The centrifuge bottles were loaded in opposing sides of the bucket and were ballasted appropriately to achieve balance when spinning.
- The spinning cycle consisted of
 - Ramp-up to 3000 rpm over 1 min,
 - Hold at 3000 rpm for the specified amount of time (typically 5 min), and
 - Ramp-down from 3000 to 0 rpm over 2.5 min
- Specimens were then weighed after each spinning cycle at 5-min intervals from 5 to 30 min and at 90 min. Between each cycle, the specimens were wrapped in a new Chemwipe to aid in the absorption of resin that had been driven out.

2.3 Sonication Processing

Four of the printed specimens were processed using sonication, performed in a Branson Ultrasonic Cleaner Model 5510R-DTH (Branson Ultrasonics, Danbury, Connecticut). Machine output is rated at 135 W with a frequency of 42 kHz \pm 6%. The ultrasonic bath was filled with distilled water to a level sufficient to exceed the height of the containers holding each specimen (Fig. 6).

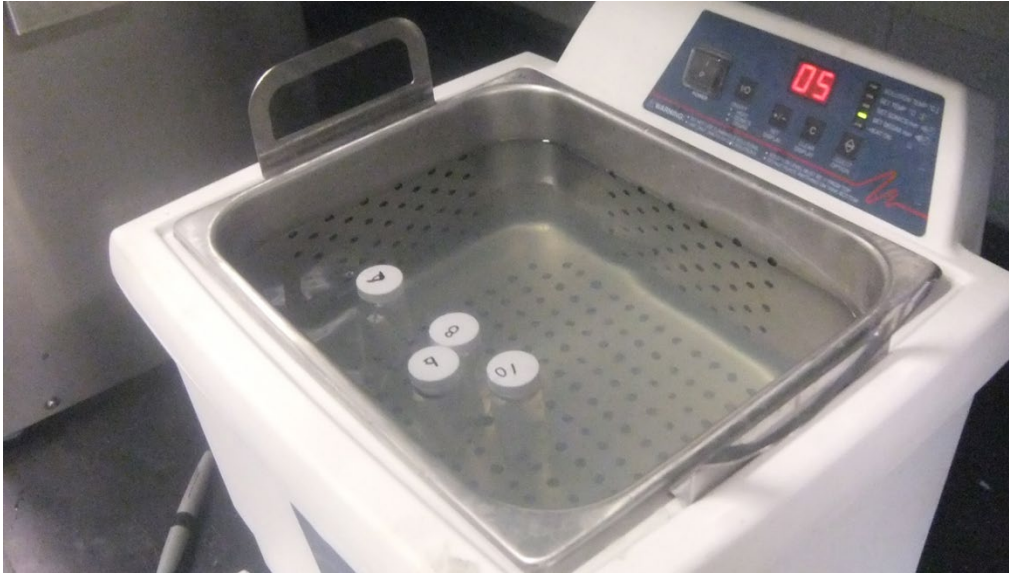


Fig. 6 Sonicator bath with glass vials containing each specimen

The sonication process consisted of the following:

- Each individual sample was placed in a cylindrical glass container, which was filled with Dowanol tripropylene glycol methyl ether (TPM) solvent and capped.
- Specimens were placed in the sonicator water bath at room temperature. Sonication occurred at a frequency of 42 kHz. The specimens were removed at specified intervals, dried under pressurized air, weighed, and returned to the container for additional sonication. Weighing occurred over 5-min time intervals from 5 to 45 min, and again at 105 min.

3. Results

3.1 Centrifuge Processing

Figure 7 shows the effect of centrifuge time on the mass of the specimen, where a reduction in mass is attributed to the removal of entrapped resin from the interior of the specimen. It is apparent that the greatest amount of resin was removed in the first 10 min of processing. Small amounts of resin are removed during the next 10 min of processing. Beyond 20 min, the sample reached a near steady-state, indicating that most of the resin that could be removed by this technique had been removed. Samples were observed after every processing cycle. Visual inspection suggested that each centrifuge cycle cleared resin out from a number of pores in the structure of the sample, with the most removal occurring after the first centrifuge

cycle. Aggregations of resin could be found stuck between the Chemwipe and the porous wall of the specimen facing the Chemwipe after each cycle.

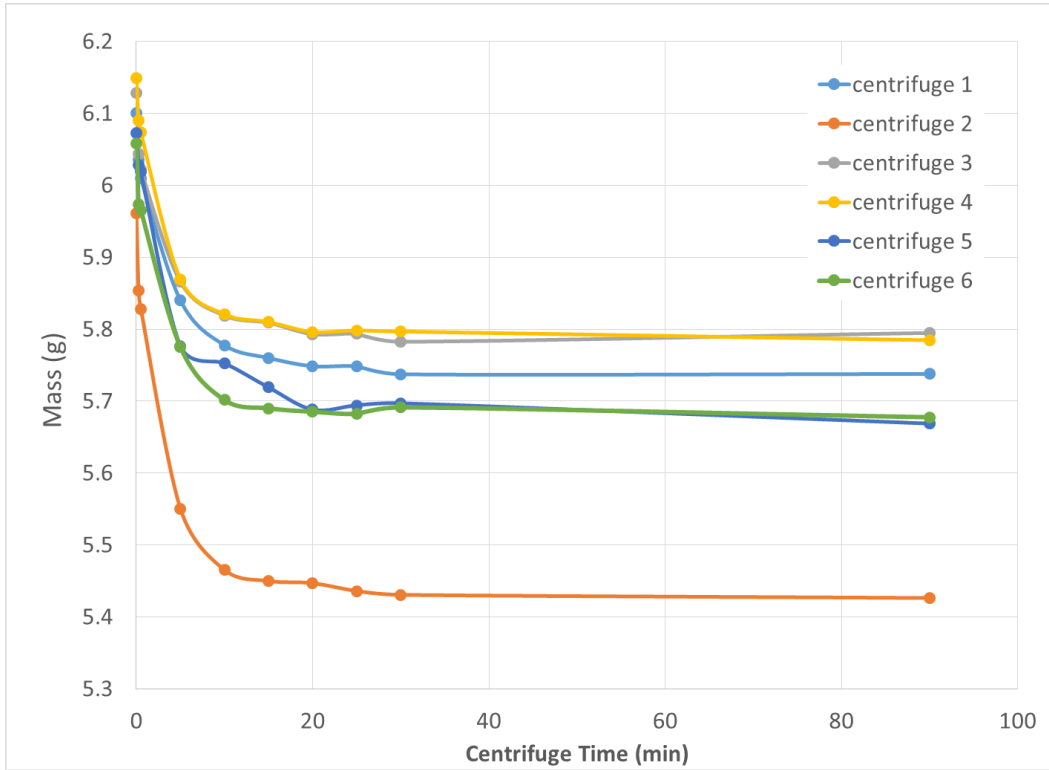


Fig. 7 Sample mass as a function of centrifuge processing time

3.2 Sonication Processing

Figure 8 shows the effect of sonication time on the mass of the specimen, where a reduction in mass is attributed to the removal of entrapped resin from the interior of the specimen. The greatest removal of resin occurred in the first 20 min of processing, though not as rapidly as that observed for centrifuge processing.

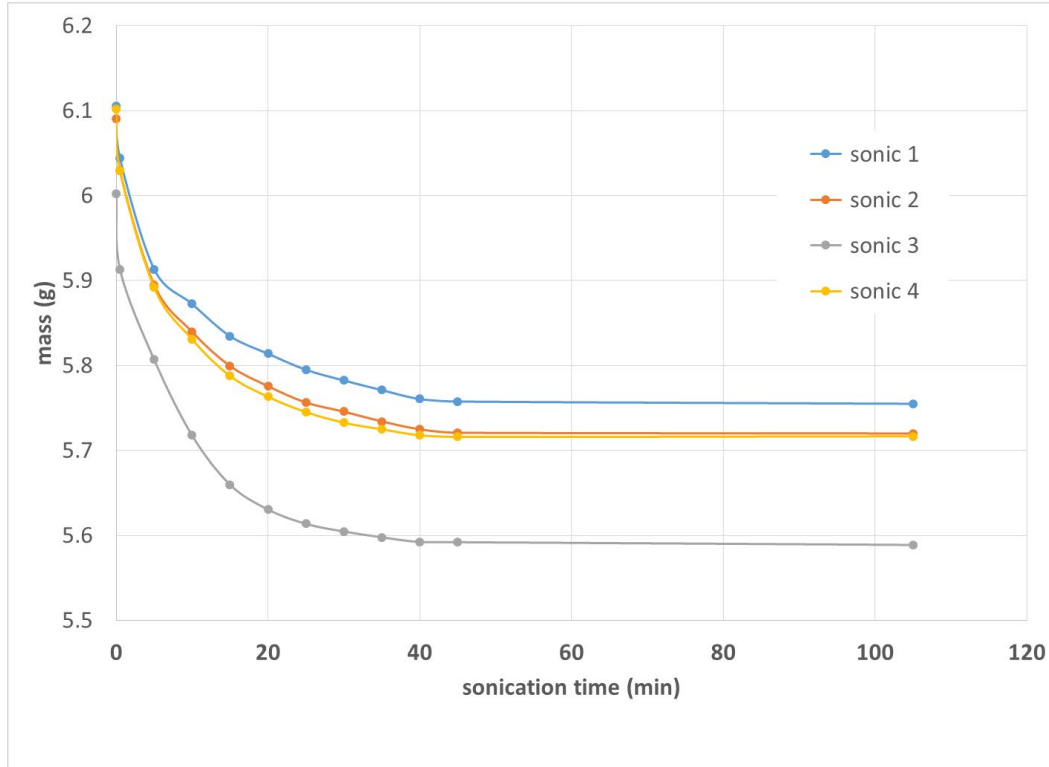


Fig. 8 Sample mass as a function of sonication processing time

4. Discussion and Conclusions

This study investigated the postprocessing of SLA manufactured parts by centrifuge and sonication treatments to remove entrapped resin from a porous internal structure. Both techniques were successful in removing entrapped resin. The most resin was removed with the sonication technique, as measured by total weight lost after all the processing was complete (Fig. 9). However, when considering weight lost from more than 5-min intervals, the centrifuge processing achieved a greater resin weight loss earlier in the process. The most resin was removed after the first 5-min spinning cycle. The centrifuge process had reached a steady state by about 30 min of spinning. Beyond that time, very little additional resin was extracted. A similar steady state was achieved after about 45 min for the sonication process.

These results indicate that if not restrained by time, sonication may remove slightly more resin than centrifuge processing. However, if constrained by time, the centrifuge can remove more resin in a shorter period of time, especially over the first 5 min of spinning. Future studies may seek to quantify the combination of centrifuge and sonication postprocessing techniques to achieve even better results in extracting entrapped resin.

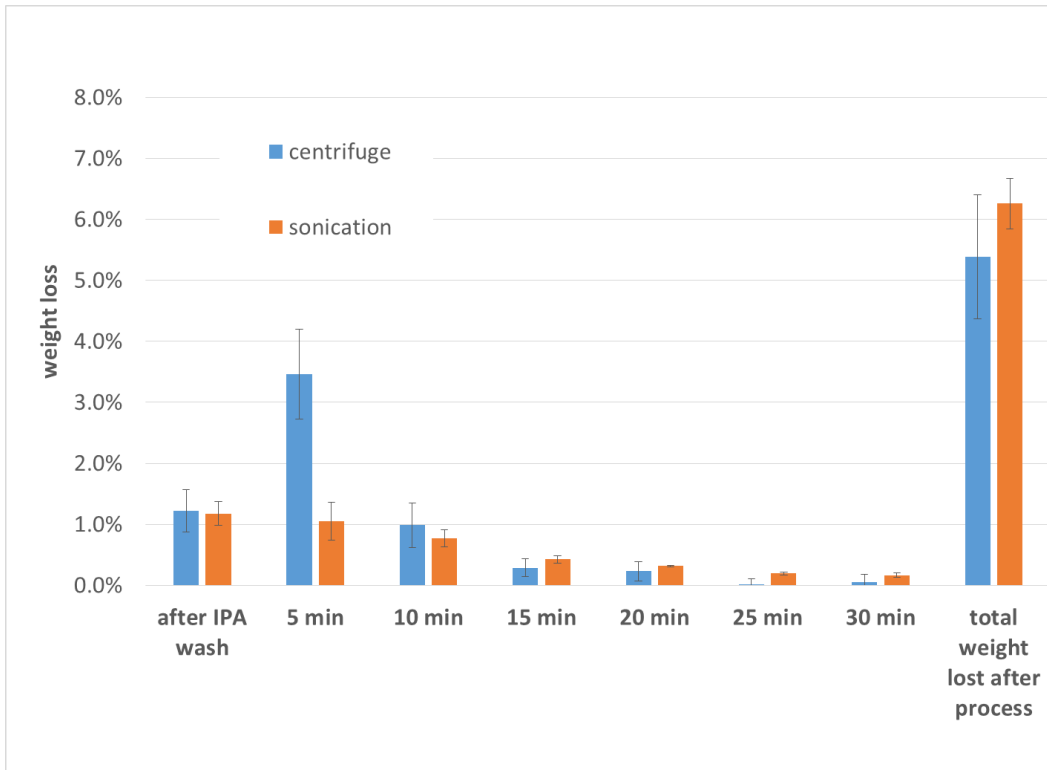


Fig. 9 Percentage weight reduction from one cycle of processing to the next, averaged over each postprocessing technique. Error bars show \pm standard deviation.

5. References

Gardner JM, Toal PM, Plaisted TA, Beitzel DD, Wetzel ED. A material simulant for replicating the impact response of playing field surfaces. Proceedings of the Institution of Mechanical Engineers, Part P: Journal of Sports Engineering and Technology; 2017 Aug 10. doi: 10.1177/1754337117723756.

List of Symbols, Abbreviations, and Acronyms

AM	additive manufacturing
IPA	isopropanol
SLA	stereolithography
UV	ultraviolet

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