Direct Conversion of McDonald's Waste Cooking Oil into a Biodegradable High-Resolution 3D-Printing Resin

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Ⓢ *Supporting Information*

ABSTRACT: Used cooking oil disposal is a concern at the global scale. Direct disposal from domestic households and restaurants via drains can result in serious environmental issues. In this study, waste cooking oil was collected directly from vats in a McDonald's restaurant and acrylated via a straightforward one-step reaction. After the addition of a photoinitiator and without any photoinhibitor, the product can be printed using a commercial 3D printer. The formulated resin produced high-resolution prints with features down to 100 micrometers. The rapid prototyped prints show considerable thermomechanical stability, morphological homogeneity, and biodegradability when compared to a state-of-the-art research resin and a commercial resin. This paper introduces the concept that waste cooking oil can be directly converted into a high-value commercial 3D-printing resin, which may have considerable societal benefits including reduction of waste and carbon emissions.

KEYWORDS: *waste cooking oil, one-pot reaction, 3D-printing resin, biodegradable, UV curable*
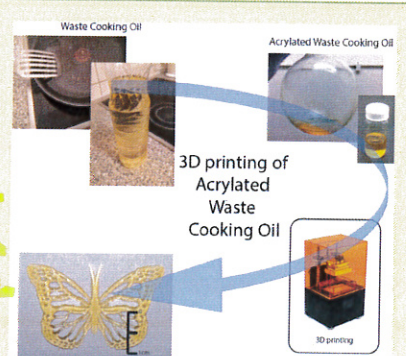
INTRODUCTION

Since its inception, 3D printing has revolutionized the design and manufacture of new products. It is currently being used to fabricate physical models for early concept verification (e.g., form, fit, and function), as well as reducing lead times for product development.[1] 3D printing offers multiple advantages over traditional manufacturing techniques, including reduced material waste, lower energy utilization, and the formation of structures not possible with traditional manufacturing processes.[2] In recent years, the popularity of the photopolymerization-based stereolithographic approach (SLA) and digital light projection (DLP) has increased exponentially.[3] These two technologies use UV-induced radical polymerization to cure the printed resin and to construct 3D objects either layer-by-layer or in a continuous fashion. Advantages over more traditional fused deposition modeling (FDM) include higher resolution, smoother surfaces, increased z-axis strength, and faster manufacturing. However, the significant costs of high-resolution photocurable resins used in SLA/DLP (can exceed $500/L) are an obstacle to widespread application.[4]

To resolve this problem, several studies have explored the possibility of synthesizing resins from low-cost materials[5,6] using simple and economic processes.[7,8] Almost all the commercial SLA/DLP resins presently on the market are based on acrylate or epoxide monomers, which can be synthesized from unsaturated compounds.[9] Soybean oil (the most inexpensive, mass-produced, unsaturated vegetable oil)

has therefore attracted attention as a potential source for making SLA/DLP resins. The first reported study on the epoxidation of soybean oil was performed by Park et al.[10] who used amberites as catalysts to react soybean oil with acetic acid and hydrogen peroxide. Campanella et al. further improved the reaction yield by replacing the amberites with amorphous Ti/SiO2.[11] Based on epoxidized soybean oil, Behera and Banthia successfully synthesized acrylated soybean oil by utilizing catalytic triphenylphosphine.[12] More recently, Zhang et al. disclosed a novel one-step synthesis of acrylated soybean oil, which greatly reduces the cost and time of making these types of materials.[13,14] By incorporating vanillin dimethacrylates into the formula, Lebedevaite et al. synthesized a photoinitiator-free 3D-printing resin.[15] This is important as it reduces the complexity of the formulations and can eliminate the need for the addition of dyes which can become environmental contaminants. All these studies show considerable potential for converting low-cost materials into useable 3D-printing resins.

Waste cooking oil (WCO) refers to oils and fats used for cooking or frying food substances at the household, commercial, and industrial levels. The direct discharge of WCO into the sewage system often clogs sewage lines due to the build-up of fats, whereas recycling can help local

communities to reduce their carbon footprints and provide a form of revenue.[16] Global production of WCO is considerable and growing rapidly.[17] Currently, China is the single largest producer of WCO (500 million tons in 2011),[16] in comparison to India,[18] the European Union,[19] and the United States.[20] Fast food chains are significant consumers of cooking oil. As the world's largest fast food chain, with more than 10% of the global market share,[21] McDonald's is estimated to produce more than 600 tons of WCO per day.[22] Every year, it costs millions of dollars for fast food restaurants to process its waste, and the majority of these expenses go toward WCO-related processes. The market for recycling WCO is still relatively young, yet is large and fast-growing. The cost of collecting and transporting WCO in France alone was estimated to be 540 million dollars in 2014.[23] Most recycled WCO is currently used in the production of soap and biodiesel.[24] WCO demands a price of $750–1000 per ton to large biodiesel producers, while biodiesel is often marketed at $1300 per ton.[25] However, the recent decrease in the price of oil is driving WCO-derived commodities down, making recycling via relatively cheap commodities (e.g., soap and biodiesel) less commercially attractive.[26,27] Hence, it may be transformative for recycling programs if high-value commodities can be manufactured directly from WCO. Interestingly, due to the rampant growth of heart disease in developed countries, most of the major fast food chains have adopted healthier unsaturated oils in their restaurants over the last couple of decades.[28,29] In turn, the double bonds in these unsaturated oil molecules permit acrylation with acrylic acid (AA), which allows cross-linking to take place during the UV-curing process. Therefore, it is important to assess whether WCO can be directly converted into a UV-curable resin suitable for 3D printing.

In the present study, used cooking oil was obtained directly from a McDonald's restaurant at the time of day it would normally be disposed of. A modified version of Zhang's approach is applied here to perform the acrylation of WCO.[14] After confirming the successful acrylation of the WCO, the product was formulated into a 3D-printing resin and printed by a commercial 3D printer. The resulting prints show an outstanding resolution with features down to 100 micrometers, which is comparable with prints made from a high-resolution commercial resin ($525/L). Subsequent dynamic mechanical thermal analysis (DMTA) indicates the waste cooking oil-derived prints possess a higher T_g and larger storage modulus than those derived from virgin soybean oil while having similar morphological homogeneity, which is consistent with higher cross-link density. Furthermore, tests demonstrate a superior biodegradation rate of this material in comparison to other resins. To our knowledge, no researchers have assessed whether waste cooking oil from a fast food restaurant can readily be converted into a UV-curable resin suitable for 3D printing.

MATERIALS AND METHODS

Materials. WCO was collected from a McDonald's restaurant (Toronto, Canada), directly from the cooking vats at the same time as the oil would normally be sent for disposal. Acrylic acid, boron trifluoride etherate ($BF_3 \cdot Et_2O$), $NaHCO_3$, NaCl, and $MgSO_4$ were purchased from Sigma-Aldrich. Hexane was purchased from Fisher Scientific. All the reagents were used as supplied without any further purification.

Synthesis of the Acrylated Waste Cooking Oil (AWCO). The WCO was filtered using filter paper (d = 15 cm; pore size = 20−25 μm, Whatman) to remove insoluble materials. In a typical reaction, 44

mL acrylic acid (6 equiv.), 100 mL WCO (3.3 equiv.), and 8 mL $BF_3 \cdot Et_2O$ (0.6 equiv.) were added to a three-neck 500 mL flask with a condenser with magnetic stirring. (1 equiv = 1 double-bond equivalent in WCO) The mixture was then heated to 80 °C for 4 h. The mixture was cooled to room temperature and stirred for a further 18 h. Hexane was then added (400−450 mL) to dissolve the organic components followed by washing with 5% aqueous $NaHCO_3$ and NaCl solutions to remove unreacted acrylic acid and catalyst (which can be further recycled). After washing and drying the resulting clear solution was evaporated under vacuum, and the hexane recycled for further use. Finally, around 42 mL of AWCO was obtained. According to NMR spectroscopy, the final product after optimization contained about 2.01 acrylates/molecule, resulting in a conversion rate of about 60.9% (see later). As mentioned in a previous study,[13] the addition of hydroquinone (HQ) (a free radical inhibitor) during the reaction did not change the acrylation degree significantly, so no free radical inhibitor was used in this study.

Characterizations of AWCO. ^{1}H NMR measurements were performed using an 11.7 T Bruker Avance III HD NMR spectrometer equipped with a 5 mm TCI cryoprobe. To prepare the NMR sample, the resin (0.5 mL) was dissolved in 1 mL $CDCl_3$. Spectra were acquired using a standard 1D ^{1}H pulse sequence with a recycle delay 5 × T_1, 7.4 μs 90° pulse, and 32 K time domain points. Spectra were processed using a function corresponding to 0.3 Hz line broadening in the transformed spectrum and a zero filling factor of 2.

Attenuated total reflectance−Fourier transform infrared spectroscopy (ATR-FTIR) analyses were performed on a Bruker Alpha-P FTIR spectrometer for the original WCO sample, the AWCO sample, and a 3D print using the AWCO. The spectrometer consisted of a diamond crystal ATR with a KBr beamsplitter. The scanning range of the FT-IR used for this analysis was from 4000−360 cm^{-1} with a resolution of 2 cm^{-1}. Eight scans were collected for each sample in total.

3D-Printing Process. AWCO was formulated into a 3D-printing resin in this study. 1% Irgacure 819 (Sigma-Aldrich, St. Louis) was added as a photoinitiator,[30] and no photo inhibitor was added. For comparison, two other resins were also tested. One was a state-of-the-art, biocompatible soybean-based resin developed for making 4D medical scaffolds,[31] and as described in the publication was also used without photoinhibitors. The other was a commercial resin (MiiCraft BV007A, specially developed for microfluidic devices costing $525 (USD) per liter) purchased from imakr.com and used as is. A Solus DLP 3D printer was used in this study. The x/y resolution was set at 41.7 micrometers in an 80 mm × 45 mm build area and the z-axis was incremented at 25 micrometers per layer. The exposure time (the shortest time that gave a successful print) was determined empirically for the resins by decreasing the exposure until the object failed to print. This approach guaranteed enough UV exposure for a successful print while minimizing overexposure, which can lead to loss of fine structural features. Optimal exposure times were 200 ms, for both the McDonald's and acrylated epoxidized soybean oil resins, while the MiiCraft required 300 ms. When the prints were completed, the excess resins were drained and reused. The 3D prints were then removed from the printing bed and washed with isopropanol to remove excess resin.

Characterization of 3D Prints. Dynamic mechanical thermal analysis (DMTA) was used to characterize the temperature-dependent mechanical properties of the dried samples. 5 mm × 10 mm samples were cut out of the hydrogel films, and the thickness was measured with a calibrated micrometer. The storage (E') and loss (E'') moduli of dry samples were measured under tension as a function of temperature on a TA DMA Q800 analyzer at a frequency of 1 Hz over a temperature range of −50 °C to at least 200 °C with a heating speed of 3 °C/min.

Synchrotron-based small-angle X-ray scattering (SAXS) measurements were performed on BM26B (DUBBLE) at the European Synchrotron Radiation Source, Grenoble, France. The sample-to-detector distance was 3 m using a wavelength λ = 0.9994 Å. A Dectris Pilatus 1 M detector with a resolution of 981 × 1043 pixels, and a pixel size of 172 × 172 μm was employed to record the 2D SAXS

scattering patterns. Standard corrections for sample absorption and background subtraction were performed. The data were normalized to the intensity of the incident beam (in order to correct for primary beam intensity fluctuations) and were corrected for absorption, background scattering. The scattering pattern from AgBe was used for the calibration of the wavenumber ($q = 4\pi\sin\theta/\lambda$) scale of the scattering curve. The sample was placed in a capillary (d = 2 mm) and kept at 20 °C using a Linkam stage. Five SAXS data frames were acquired for every sample, and each one was measured for 1 min (totally 5 min).

Biodegradation of these 3D prints was evaluated by soil burial tests.[32] All the specimens (10 mm × 10 mm × 2 mm) were buried in containers filled with soil (grassland in Heerlen, the Netherlands) and buried 50 mm under the soil surface. The containers were then placed in a temperature-controlled humidity chamber (CSZ Z-Plus Test Chamber, Weiss Technik UK Ltd.) kept at 30% relative humidity and 25 °C for 14 days. After the tests, all specimens were washed, cleaned, and dried under vacuum at 25 °C for 24 h. The weight of the samples were measured before (W_{before}) and after (W_{after}) the biodegradation test, and the weight loss calculated by ($W_{before} - W_{after}$)/W_{before}.

■ RESULTS AND DISCUSSION

As mentioned above, a simple one-step Michael-addition-type reaction was employed here to perform the acrylation of waste cooking oil while using $BF_3\cdot Et_2O$ as the catalyst (for details, see Materials and Methods). Optimization of the synthesis is discussed later in the main paper.

Structural Characterization of AWCO. The WCO and reaction product (AWCO) were analyzed by ^1H NMR spectroscopy. Figure 1b compares unreacted WCO (Figure 1b.I) with WCO after acrylation (Figure 1b.II). The corresponding NMR assignments were performed in accordance with previous studies[13] with the alpha carbonyl methylene resonance (H_i, Figure 1a) used as an internal reference. By analyzing these spectra, information about the WCO and its conversion to acrylates can be calculated. The triglyceride content of the WCO mixture can be confirmed via the ratio between the peak area of H_i and H_g in WCO (Figure 1b.I), which was found to be approximately 6:4. This is the ratio expected for oil made up purely of triglycerides. This suggests that McDonald's either (i) uses oils with very high triglyceride content or (ii) other components present in the oils soak into the food, leaving only triglycerides in the waste.

The alkene double-bond content in the WCO can be determined by the following equation:[11]

$$\text{Double bonds per WCO molecule} = \frac{(A_{d,e} - A_g/4)/2}{A_i/6} \tag{1}$$

where A is the integrated area under the corresponding peaks. The McDonald's WCO (Figure 1b.I) contains an average number of alkene double bonds per molecule of around 3.30, which is lower than that reported for pure soybean oil,[13] and consistent with McDonald's using a blend of different oils.[33] Importantly, the extent of the acrylation reaction can be determined by comparing the peak area of H_a and H_i in the spectra of AWCO using the following equation:[11]

$$\text{Acrylate groups per AWCO} = \frac{A_a}{A_i/6} \tag{2}$$

The average number of acrylate groups per triglyceride of the AWCO sample, when using the optimized conditions of Zhang et al.[13] (Figure 1b -II), was found to be 2.01. On the basis of the average double-bond number calculated above in
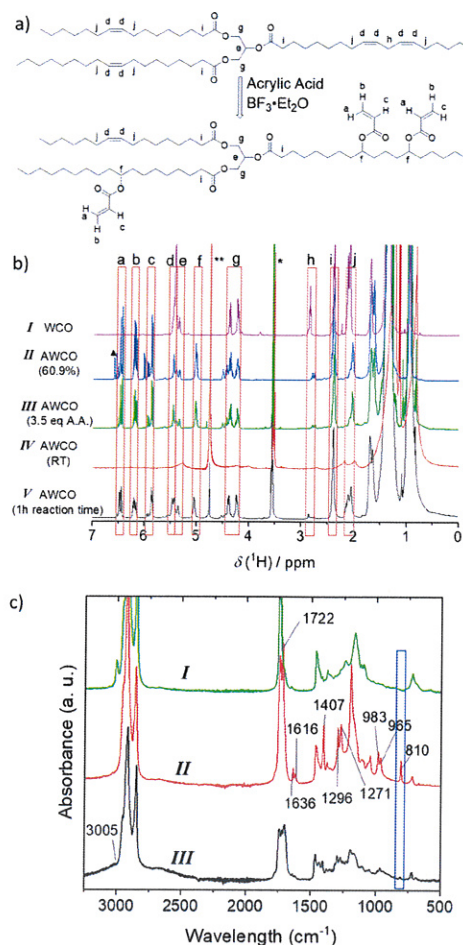

Figure 1. (a) Synthetic scheme of AWCO from WCO, AA, and $BF_3\cdot Et_2O$; and (b) ^1H NMR spectra of WCO and AWCO. (*: Diethyl ether; **: H_2O; ▲: product from self-Michael addition). I: McDonald's WCO; II: conditions optimized by Zhang et al.;[13] III: conditions with less acrylic acid; IV: reaction at room temperature; V: AWCO reaction time of only 1 h; (c) FT-IR spectra of I = WCO; II = AWCO; III = resin after UV curing.

the original WCO, the calculated conversion of double bonds to acrylates was 60.9%. This result is even better than the reported value for pure soybean oil under the same condition[13] and indicates that WCO has considerable potential for conversion into a highly acrylated UV-curable resin. The formation of acrylates is also supported by FT-IR analyses (Figure 1c). The characteristic C−H stretching band (~3005 cm^{-1}) of C=C−H in WCO disappeared after the acrylation reaction, while a series of peaks attributed to C=O (vibration mode: 1722 cm^{-1}), CH=CH$_2$ (C=C vibration mode: 1636 cm^{-1}, 1616 cm^{-1}; CH$_2$ scissoring vibration mode in CH$_2$=C: 1407 cm^{-1}; CH vibration in acrylate CH=C: 1296 cm^{-1}, 1272 cm^{-1}; CH$_2$ rocking vibration in CH$_2$=C: 966 cm^{-1}; CH bending deformation in CH=C: 810 cm^{-1})[34,35] appeared in the spectrum for the AWCO.

Optimization of the Acrylation Reaction of WCO. A series of optimizations were carried out to see if the reaction could be made "greener" and the resin synthesized in a more cost- and energy-efficient manner. As shown in Figure 1b and Table 1, different proportions of the WCO to AA were tested when n(WCO) was kept at 100 mL. An example is shown in

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Table 1. Double-Bond Conversion Rate for Acrylation Reactions under Different Conditions[a]

BF$_3$·Et$_2$O (equiv)[b]	AA (equiv)[b]	reaction time (h)	reaction temperature (°C)	double-bond conversion rate (%)
0.2	6	4	70	33.4
0.6	6	4	70	60.9
1	6	4	70	48.2
0.6	3.5	4	70	39.1
0.6	9	4	70	42.0
0.6	6	1	70	34.8
0.6	6	4	20	0
0.6	6	4	100	0

[a]18 g WCO (20 mL, 20 mmol, 4 equiv.) was used in every case. [b]1 equiv = 1 equiv double bond.

Figure 1b.III where the ratio of AA was reduced to 3.5 equiv. This leads the double-bond conversion rate to decrease to 39.1% (compared to 60.9% for the optimized reaction). In addition, the acrylic acid ratio was also increased to 9 equiv, but the double-bond conversion rate was also reduced. This is likely a result of the AA molecules undergoing self-Michael addition reactions,[36] which has been reported in the acrylation of soybean oil.[13] It is worth noting that this self-Michael addition reaction also occurred to a small extent with 6 equiv AA, as demonstrated in the NMR analyses (see small peak around 6.6 ppm (triangle up solid)). Attempts were additionally made to reduce the reaction time and determine if the reaction would proceed at room temperature (both of which could save energy and make recycling WCO in this manner more attractive). Unfortunately, the reaction failed to proceed at room temperature (see Figure 1b.IV and Table 1), and the conversion of double bonds to acrylate was reduced with shorter reaction times. As such, it was concluded that the optimal reaction conditions for acrylation of McDonald's

WCO are 70 °C for 4 h for a mixture of 6 equiv HA, 3.3 equiv WCO, and 0.6 equiv BF$_3$·Et$_2$O. The reproducibility of the synthesis is shown in Figure S4. This is very similar to the conclusion made for the acrylation of pure soybean oil.[13] It can be explained through the composition of McDonald's WCO, which is essentially described as a mixture of canola oil, corn oil, and soybean oil.[33]

3D-Printing Tests. In this study, a butterfly design containing a range of larger and smaller structural features was selected (for the full details, see the Materials and Methods). A digital rendering of this 3D model is shown in Figure 2a. Figure 2c displays the printed WCO with detailed features down to ~100 micrometer resolution, which approaches the performance of some of the most expensive commercial resins (for detailed image analyses see the Supporting Information) but not as high as optically optimized research resins, which were recently reported to produce features <100 micrometers.[30] This excellent printing is performed without the addition of a photoinhibitor. Photo-inhibitors are commonly used in commercial resins to block stray light and ideally restrict curing to only where the light is focused. If photoinhibitors are not used, the light will usually spread out during irradiation, and all features can be lost.[37] This was found not to be the case for the AWCO, and excellent resolution was observed without any photoinhibitor present. This indicates that some pigments in the AWCO (possibly generated during the cooking process from the food[38]) absorb the UV light during the curing and localize the curing without additional additives. This is particularly attractive as an environmentally friendly product since it removes the need for adding dyes, which can be costly and act as environmental contaminants.[39]

For comparison, an acrylated epoxidized soybean oil (AESO, without a photoinhibitor) as per the original publication[31] was also tested. The AESO resin had a tendency to overcure due to
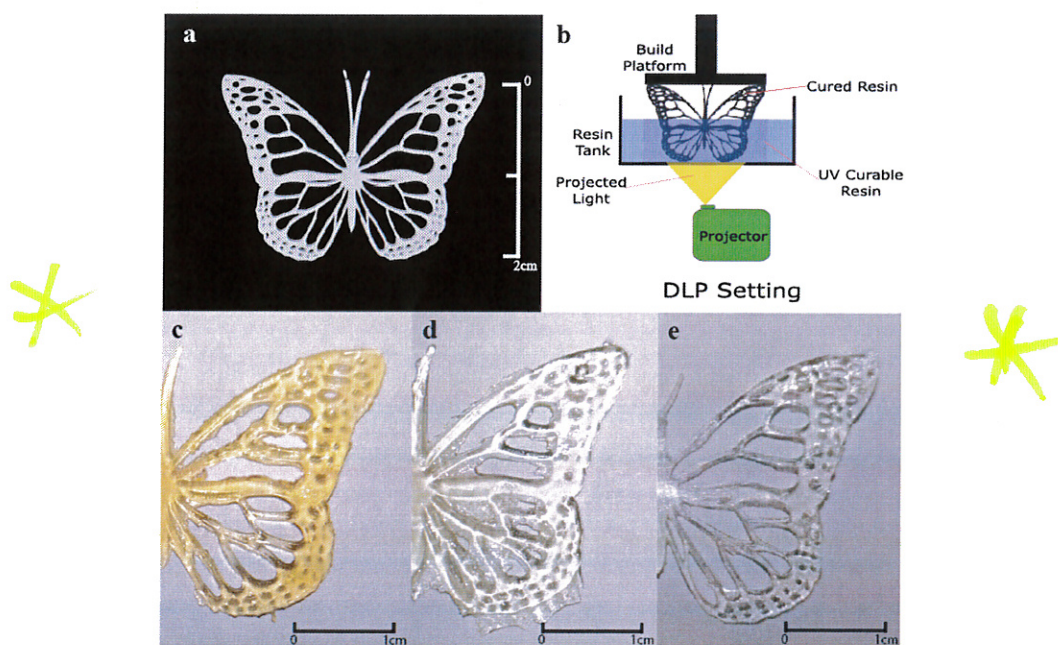

Figure 2. (a) 3D butterfly digital model; (b) simplified DLP 3D printing scheme used in this study; 3D-printed butterfly from (c) McDonald's AWCO, (d) acrylated epoxidized soybean oil, and (e) commercial resin MiiCraft.

the diffraction and spreading of the light. As a result, some of the open spaces in the butterfly design were filled in with cured resin, and the structural definition was reduced in comparison to the resin derived from the AWCO (Figure 2d).

Furthermore, an expensive ($525/L) high-resolution Mii-Craft commercial resin was also compared. From the authors' combined experiences, the BV007A resin is the highest resolution commercial DLP resin on the market. Interestingly, the resin derived from the AWCO printed nearly as well. Figure 2e shows that the MiiCraft print has a slightly improved resolution in comparison to the AWCO; however, some features were lost (e.g., antenna was shortened in the MiiCraft print) even after the optimization. A detailed statistical analysis between the computer model and the 3D prints is provided in the Supporting Information (see Table S1). Surprisingly, the resin created from the AWCO produced a print improved over a state-of-the-art resin developed for medical research (AESO) and nearly as good as the highest resolution commercial 3D-printing resin (based on the authors' experience) on the market (MiiCraft).

Characterization of 3D Prints. Since they have rather similar chemical composition, the prints made from both AWCO and AESO resins were further analyzed by dynamic mechanical thermal analyses (DMTA) and small angle X-ray scattering (SAXS). As shown in Figure 3a, a lower glass
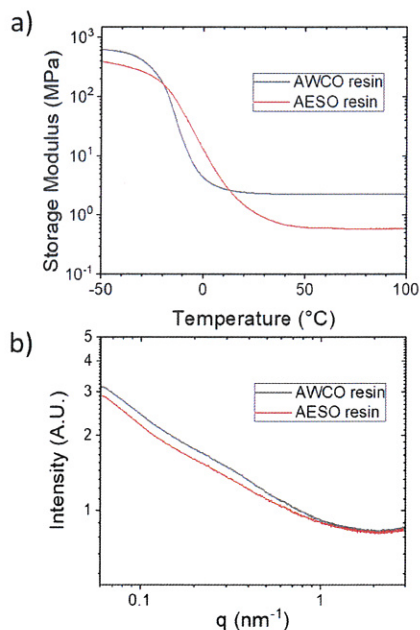

Figure 3. (a) Storage modulus of AWO and AESO cured resins between 0 and 150 °C in DMTA analyses and (b) SAXS profiles of AWO and AESO cured resins at 20 °C.

transition temperature T_g (−10 °C) can be found from the DMTA analyses of this cured AWCO resin as compared to the prints made from the AESO (1 °C) whose value is similar to the one in the previous study.[40] The difference may be explained by the presence of free fats or pigments generated during the cooking process, which may act as plasticizers reducing the brittleness of the polymer network.[41−43] Furthermore, a reduction in the peak height of the tan σ curve for AWCO prints (Figure S3) and a higher storage modulus after T_g for AWCO prints support a higher cross-link

density and "rubbery" strength, which supports improved thermomechanical performance of the prints at room temperature.[42,44] SAXS analyses (Figure 3) show similar spatial correlation of electron density for both resins over the length from 5 to 100 nm, and no distinguishable morphological domains can be observed, which is consistent with a previous study of the AESO resin.[45] This demonstrates that the two cured resins achieved similar morphological domain distribution at the nanometer scale. This in turn implies that any free fats or pigments from cooking in the AWCO do not affect the curing process, other than potentially acting as a photo-inhibitor, which improves resolution as mentioned above.

Biodegradation Test. The biodegradability of these prints was also measured by calculating the weight loss of these samples in soil burial tests over 14 days. As shown in Figure 4,
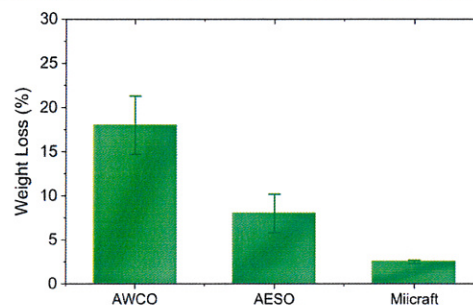

Figure 4. Biodegradability test of all the 3D prints from different resins that are measured by the weight loss of the prints after being placed in a humidity chamber for 14 days.

a large weight loss (~25%) can be found for AWCO, which is significantly higher than other samples. This can be explained by the presence of bioavailable fat molecules (or microscopic food particles left over from cooking) in the AWCO prints, which themselves can be rapidly biodegraded[46] and in turn give better access of the polymer network.[47] Physical accessibility is known to be a key factor determining the biodegradation of natural polymers in soil.[48,49] The rapid biodegradation of the AWCO prints is particularly interesting and suggests that the printed waste oil materials should degrade over time in a soil environment and thus turning a problematic waste stream into a useful commodity that is ultimately also environmentally friendly over longer time periods.

■ CONCLUSIONS

In summary, a biodegradable high-resolution 3D-printing resin was created through a one-step reaction from McDonald's WCO. After several optimizations, the printed 3D model from this AWCO resin displayed excellent printable resolution, which was nearly as high as the leading DLP resin on the market. In addition, the thermomechanical and morphological properties of the AWCO resin were similar or better than a state-of-the-art soybean oil-based resin specifically developed for 4D tissue engineering. Furthermore, the considerably-improved biodegradability of the AWCO resin in soil suggests that products made from waste cooking oil could also be environmentally friendly. Given that high-resolution DLP resins are sold at a premium price, the approach of converting a WCO to a high-value commodity is very promising. This is particularly attractive as the financial barriers currently

associated with recycling WCO can be removed, making the practice more widespread. Moreover, the properties of the waste cooking oil negate the need for a photoinhibitor, meaning that after acrylation, no photoinitiator (dyes), which can become environmental pollutants, is required for formulating the 3D-printed resin. The ability of WCO to produce prints with resolutions comparable to that of commercial resins is somewhat unexpected, and additional research should be undertaken regarding the recycling of WCO into curable resins. However, readers should note that while the oil represents the oil from cooking vats at a McDonald's restaurant, it represents only one store and one brand. While the conversion relies only on the presence of double bonds and therefore should be successful for any highly unsaturated cooking oils, future work is required to see how differences between stores and differences between brands may impact the recycling of waste oil into UV-curable resins. We hope this study will stimulate future research into the conversion of waste oil into high-value UV-curable commodities, which hold both economical potential for the companies and potential positive environmental impacts.

■ ASSOCIATED CONTENT

ⓢ Supporting Information

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acssuschemeng.9b06281.

> Detailed NMR assignment of WCO and AWCO, tan delta of AWO and AESO from DMTA analyses, triplicate NMR, and image analysis (PDF)

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Notes

The authors declare no competing financial interest.

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