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3D Printing of Short-Fiber Composites as an Effective Tool for Undergraduate Education in Composite Materials

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3D Printing of Short-Fiber Composites as an Effective Tool for Undergraduate Education in Composite Materials

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Abstract

Fiber-reinforced composite materials enjoy widespread uses as structural materials in myriad of modern-day applications including airframes, high-performance vehicles, consumer sports equipment, biomedical prosthetics, and building construction. Despite years of fruitful progress in the materials aspect of composite materials, the still-heavy reliance on manual fabrication and the lack of automated composite-making techniques have kept composite materials from being a high-volume production materials-of-choice and from being easily made into complex shapes with consistent quality. To this end, three-dimensional printing of composite materials—a nascent and potentially game-changing composite manufacturing technology in its own right—offers an enabling technological solutions. The work presented here details a collaborative research effort between students and faculty of Canada College and San Francisco State University (SFSU), supported by a Department of Education grant, in realizing 3D printing of short-fiber UV-curable polymer composite. Four Canada College students working alongside an SFSU student mentor, successfully designed, prototyped and commissioned an innovative extrusion mechanism capable of printing short-fiber infused polymer composites, at a single-line resolution of 0.5mm and in a consistent layer-by-layer fashion. The extrusion mechanism is capable of extruding UV-sensitive polymer that incorporates carbon fibers (7µm diameter, up to 0.1g) and cloisites nanoclay (up to 0.075g) per 1mL of the UV curable polymer, Vorex™. Various composite test specimens were printed for mechanical testing and for characterization using a scanning electron microscope. Results arising from this research point to: (i) mechanically robust short-fiber composites that are capable of being produced by direct 3D printing, and (ii) a remarkable dispersion of short carbon fibers in the polymer matrix, which displays relatively defect-free interfacial bonding. Through a 10-week theoretically grounded, hands on undergraduate research experience, the community college students were able to deepen their understanding of the mechanics and manufacturing of composite materials, starting from scratch and against a steep learning curve, via meaningful experimentations, relentless trouble-shooting, and constant consultation with suppliers and industry experts.

1. Introduction

Fiber-reinforced composite materials have gained popularity as a structural members in aerospace, automotive, construction, transportation, biomedical, consumer sports equipment industries because of their high strength-to-weight and high modulus-to-weight ratios. Fiber-reinforced composites are generally classified into two broad categories: long fiber-reinforced composites (fiber length-to-diameter ratio between 200 to 500) and short fiber-reinforced composites (fiber length-to-diameter ratio of between 20-60). The former is usually formed by embedding long, unidirectional fibers or woven cloth in a polymer matrix; while the latter by the dispersion of short fibers in a polymer matrix. The major fibers that are widely used are E-glass, S-glass, carbon, graphite, and aramid fibers; whereas common matrices range from polyester, vinyl ester, to epoxy resin. Long fiber-reinforced composites have been the mainstay of fiber-reinforced composites predominantly due to its superior strength-to-weight properties that are significantly higher than metals. Nevertheless, where significant strength-to-weight improvement is not critical relative to manufacturing cost, short fiber-reinforced composite materials are often more desirable[1]. The manufacturing of basic, small-volume short fiber-reinforced composite components are labor-intensive, generally involving a "hand lay-up" where fiber mats are laid out onto molds and coated in the polymer, followed by a thermal curing process. For large-volume production, a rich repertoire of semi-automated processes are used, which include spray-up, compression molding, thermoset compression molding, thermoplastic compression molding, injection molding, thermoplastic injection molding, and thermoset injection molding[2]. Nevertheless, the capital investment and technical complexity of these mass production techniques are prohibitive to small- and medium-sized enterprises and individuals. To date, it remains a challenge to produce customized, complex-shaped composites part, especially at the millimeter and centimeter length scales.

This work describes a collaborative summer research effort between two institutions, Canada College (a 2-year community in Northern California) and San Francisco State University (a four-year university in Northern California) in researching and developing a 3D printing method that enables the direct printing of short fiber-infused photopolymer composites. The overarching goals of the project are several: (i) to explore 3D printing as an enabling technology to realize small-volume or one-off, highly customized fabrication of complex-shaped and centimeter-scale composite structures, and (ii) to facilitate learning of key topics in mechanical engineering, including mechanics of composite materials, mechanical design, 3D modeling, basic electronics programming, and hands on fabrication. Equally important, we would like to infuse hands on tinkering into important mechanical engineering topics as a means to encourage deep learning, critical thinking, and problem solving. By having students work alongside a graduate student mentor and a faculty mentor, the additional objective is to provide the context through which minority community college students can understand how research is conducted and critical soft skills (e.g. resilience, resourcefulness, good communication) that benefit a researcher.

The faculty mentors and student mentor designed the schedule and milestones for the project, which was discussed with the team and agreed upon. A team leader was elected and was responsible for tasks assignment. During the 10 weeks, the team of undergraduate students was required to meet with the student mentor at least twice per week as a group, and more frequently on an individual basis for a "check-in", progress monitoring, and discussion of the next phase of the project. The student mentor act more like an "operations advisor" whose role is to provide regular, on-site technical advice and guidance on 3D solid modeling, hands on fabrication, and operations of the 3D printers. The faculty mentor meets on a weekly basis with the whole team, including the student mentor. For this particular project, the faculty mentor conducted several short lectures on composite materials, mechanics of solids, and basic materials science to enable the undergraduate students to be acquainted with concepts they would apply in the project.

2. Extruder System Design

The core of this work is premised on the ability to 3D print short fiber-infused UV curable photopolymer resin composites. A critical aspect of this work is therefore the design and build of an extruder system capable of simultaneously printing and curing the photopolymer. Almost all of the current dispensing systems used in 3D printing are designed to extrude highly viscous, thermoplastic. Currently, there is no known system that readily dispenses UV-curable polymer mixed with short fibers. As such, the initial objective of the project is to redesign an existing extrusion system to be able to extrude fiber-laden, UV curable polymer. The dispensing system consists of two integral components: (i) an extruder, and (ii) a UV curing system, described in the following subsections.

2.1. Extruder Design

Almost all low-to-mid range 3D printers (i.e. 3D printers with unit prices below $3000) use a thermal extruder where thermoplastic filament is fed into a heated chamber, melted, and extruded through the print head. Filaments are typically pre-fabricated into a spool and have diameters of either 1.75mm or 3mm typically. For this project, the print material feedstock exists in the form of a UV-curable, liquid photopolymer rather than a solid thermoplastic. While both types of polymers (thermoplastic vs. photopolymer) are eventually extruded, in the case of the photopolymer, a short post-extrusion UV light irradiation is necessary to fully cure the photopolymer. In addition, the extruder used in this work is required to extrude photopolymers that exhibit a range of viscosities—from the consistency of maple syrup to that of toothpaste. To this end, we used an existing 3D printer platform, but completely redesigned its extruder system. Figure 1 shows the redesigned extruder system that contains a stepper motor, an aluminum frame, a motor-driven plunger, 50mL extruder syringe with 14-gauge needle, UV LED system, and a fan. Since we are not extruding conventional thermoplastic, a heater is not part of the redesigned system. The conventional thermoplastic filament system that takes in and extrude solid thermoplastic is also replaced by a motor-driven syringe pump system that can precisely dispenses fixed volume of liquid photopolymer. Another distinct advantage of this extruder system design is that the syringe can be completely removed and replaced between prints. This feature allows syringes containing different formulations of epoxy, with or without short fibers, to be easily installed and used in printing. Aside from its versatility in printing different types of composites, the ease of changing out syringes enables the printing of layers made up of different composites.

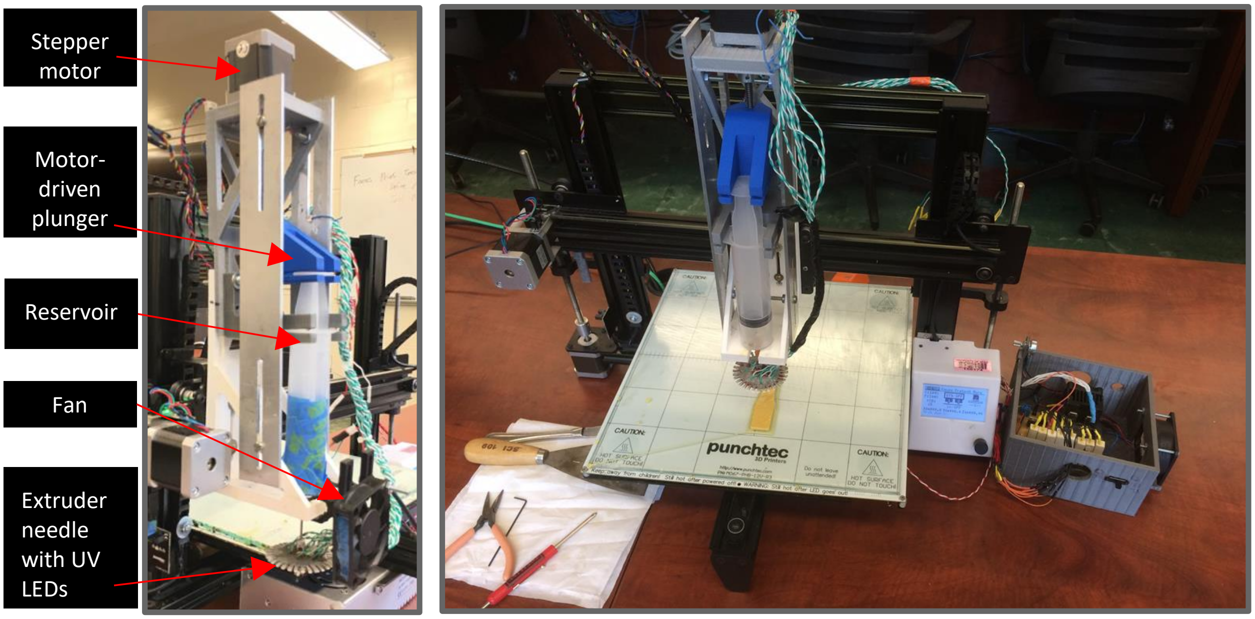
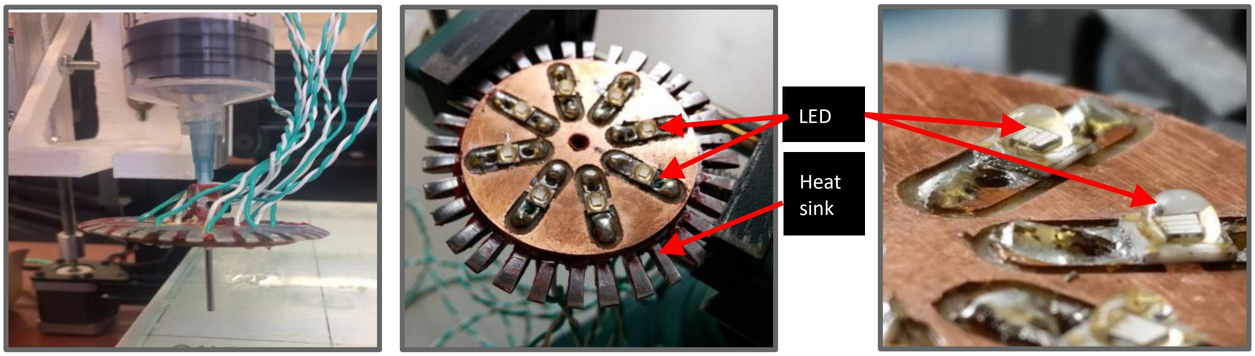


Figure 1. A newly designed extruder system (left) that is integrated with an existing 3D printing platform (right).

2.2. UV Curing System

Figure 2 shows the UV curing system that is part of the extruder needle (Figure 1). The design allows for the simultaneous dispensing and UV curing of the photopolymer. Eight 365nm-wavelength UV LEDs—each dissipating 2W—were embedded on a circular PCB board, with an aluminum heat sink to dissipate heat emanating from the LEDs. The total power output from this LED curing system is designed to achieve complete curing of the polymers used. The lack of information on the required irradiance for the polymers used compels the incorporation of a variable resistor in order to adjust the irradiance during the testing process. A circuit was designed using Eagle, a circuit design software, and milled into a printed circuit board using a desktop CNC machine, *The Other Mill*. An ATtiny85 microcontroller was programmed to send a pulse width modulation signal to the transistors and a RGB LED indicator. The RGB LED produces a color with the voltage supplied from the potentiometer: a green color meant zero volts and a purple color meant five volts were coming from the potentiometer. This assisted the process of adjusting the irradiance of the UV LEDs during the cure testing of the composites.



(a) (b) (c)

Figure 2. (a) Side view of a UV curing system that is integrated with the extruder needle. (b) Eight equally spaced 365nm UV light emitting diodes (LED) embedded in the curing system provides a quasi-isotropic lighting environment. (c) Magnified view of the embedded LEDs.

3. Materials: Fibers and Matrices

Narrowing down the matrix-fiber combination was necessary to focus on a composite mixture that provided smooth extrusion with a paste-like consistency. An ideal composite would have the ability to form a small bead when extruded and hold a shape so that more layers could be laid upon it in the printing process. The composite also needed to cure within one second of exposure to UV light to provide a completely cured composite. UV curing of resins are common in stereolithography (SLA) printing where a UV laser cures a bath of photopolymer resin layer by layer[4]. This research is adapting the curing process of photopolymers while introducing fibers in order to produce a product with higher toughness and ultimate tensile strength (UTS) when compared to traditional 3D printed materials. The mix ratio must be carefully monitored because the fiber volume fraction affects the curing of the composite. When the composite is saturated with fiber, UV light will not be able to cure the matrix because the fibers are blocking the UV light. Since the UV light is applied from the top, the fibers create a shadow in the resin, causing the shadowed part to not cure. Similarly, in random discontinuous short fiber reinforced composites, the elastic modulus and strength decrease as the volume fraction of fiber increases[3].

Several matrix materials were used, including (i) a polyester resin, Solarez (Wahoo International Inc., Vista, CA), (ii) a proprietary polyester resin, Vorex (MadeSolid Inc., Berkeley, CA) and (iii) a cationic epoxy, Loctite 3355 (Henkel, USA). All resins used were curable using a 365 nm UV light. The UV curing matrices extruded alone had a low viscosity similar to maple syrup, making them undesirable in this application. To increase viscosity a surface modified nanoclay was added to the matrix. The fibers tested for reinforcement were milled glass fibers (16 micron diameter; 230 micron mean length), Kevlar® pulp (Fibre Glast Developments Corporation), 250 milled carbon fiber (7 micron diameter; 250 micron mean length), 100 milled carbon fiber (7 micron diameter; 100 microns mean length), or silicon carbide micron-whisker (2.5 micron diameter; 50-80 micron mean length).

The mechanical properties of the composites were determined using theoretical modulus values calculated using the Halpin-Tsai model[1]. These equations predict the elastic properties of a composite material based on the geometry and orientation of the fibers and elastic properties of the matrix and fibers. Theoretical values showed that Vorex and a five percent volume fraction of 250 milled carbon fiber would provide the largest modulus. Vorex and a five percent volume fraction of 100 milled carbon fiber showed the second largest modulus.

Each batch of composite material was mixed using a spatula in a drill press running at 600 rpm from 10 to 20 minutes until the batch was visually well mixed, meaning the presence of fiber and/or clay clumps were not visible.

4. Testing Procedure

Each matrix-fiber combination was evaluated for printability and curing ability as well as print quality in order to identify the optimal parameters that would give rise to a viable composite. Testing was implemented to compare the elastic modulus and ultimate tensile strength (UTS) to that of pure matrix materials, with commercially printed 3D printed poly-lactic acid (PLA) used as a reference. The PLA samples were printed using an Ultimaker 2+ desktop 3D printer. To prepare the pure matrix tensile specimens, each matrix was poured into an identical shaped mold conforming to the dimensions of standard tensile specimen and cured with UV light. These specimens were used control specimens. The DFCs were 3D printed with the aforementioned extruder system at various volume fractions of fibers in different matrices. All tensile test specimens conform to the ASTM D638-02A standard for plastics and tensile tests were done with an Instron 3369 tensile test machine.

5. Results and Discussion

Figure 3 (a) shows that the extruder system successfully printed fully cured photopolymers—Solarez, Vorex, and Loctite 3355. Figure 3 (b) shows three as-printed specimens of Kevlar-Vorex composite and Figure 3 (c) shows post-tensile test specimens of various composites. Figure 3 (d) and 3 (e) are the scanning electron micrographs of the cross sections of a representative printed composite, E-glass/Vorex. The matrix-fiber combinations that were able to print a standard tensile test specimen are listed in Table 1. Figures 4, 5, and 6 are the tensile test results from short fiber-reinforced polymer composite using Vorex (Figure 4), Solarez (Figure 5), and Loctite 3355 (Figure 6), with 3D-printed polylactic acid (PLA) using a standard Ultimaker 2+ machine as a comparison. As expected, the test results generally show a lower elastic modulus and UTS for each composite compared to a 3D printed PLA sample. Inherently, all three polymers have lower elastic modulus than PLA when fully cured. When comparing the elastic modulus and UTS of between the molded polymer matrix specimens and its 3D printed fiber-reinforced specimens, the composites of these polymers showed lower elastic modulus and UTS than the molded polymer matrix. The key reason behind this observation is shown in Figure 3 (d) and 3 (e), where due to the non-optimized fabrication parameters, poor adhesion between layers during 3D printing leading to microcracks and delamination, as well as a significant amount of embedded air bubbles, result. Upon examining Figure 3 (e), the volume fraction of the air bubbles in our specimens are similar to or greater than the volume fraction of fibers, leading to compromised mechanical properties in the reinforced composites.

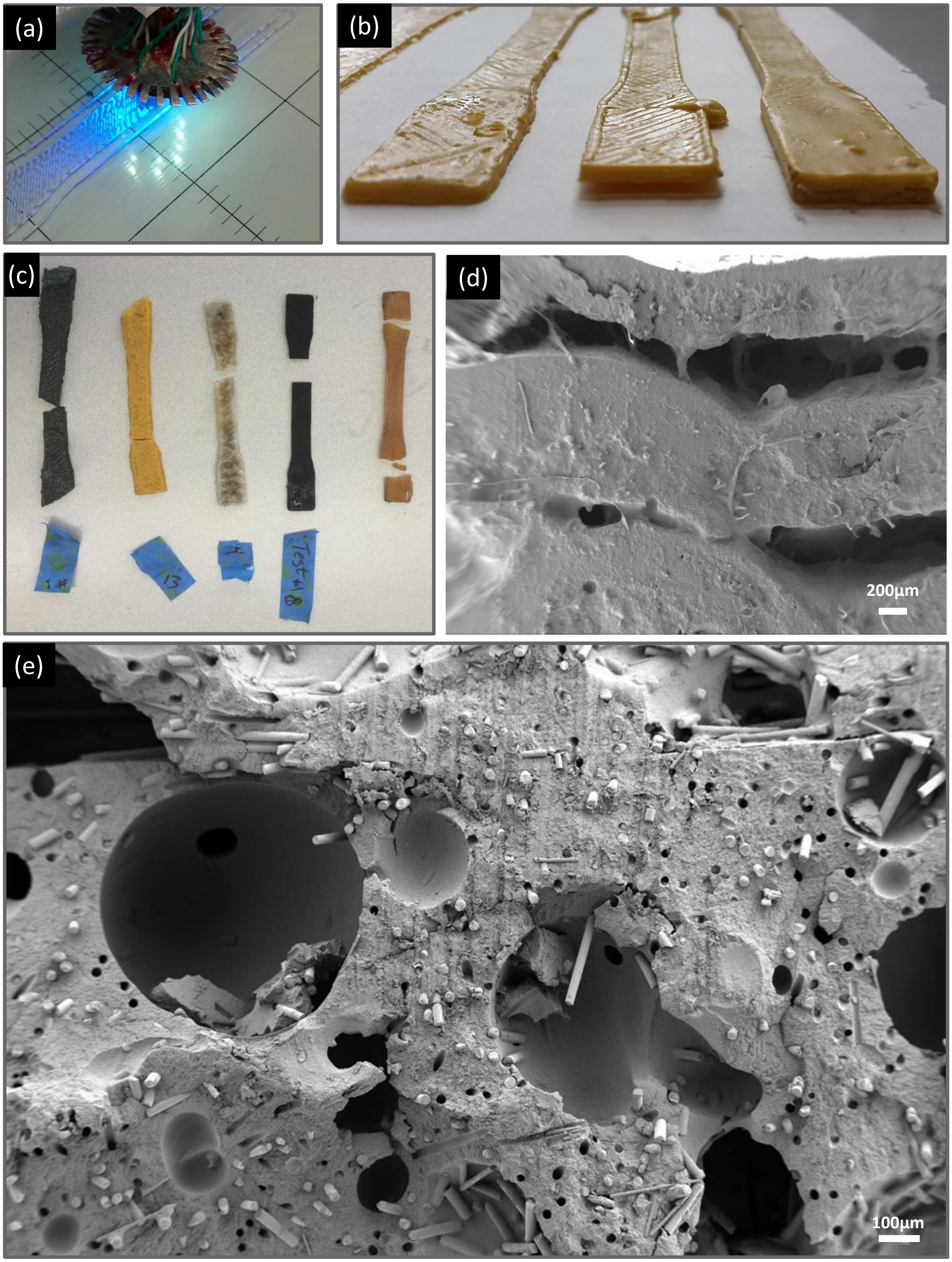

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Figure 3. (a) Printing and curing of photopolymer in progress. (b) As-printed short Kevlar fiber-reinforced Vorex composite tensile test specimens. (c) Tensile test specimens made of different combinations of fibers and polymers. (d) and (e) Cross-sectional scanning electron micrograph of a sub-optimally printed composites showing the presence of significant voids and incomplete adhesion between layers of prints.

Table 1: Recipe of each composite mixture that enabled successful printing of a standard tensile test specimen.

Specimen Index	Matrix Type	Matrix (mL)	Fiber Type	Fiber (g)	Clay Type	Clay (g)	SiC (g)
1	Vorex	80	CF 100	7.4	Nanoclay	6	3.75
2	Vorex	80	E-Glass	20	Nanoclay	10	0
4	Loctite	25	E-Glass	10	-	-	-
6	Vorex	80	CF 100	7.4	Nanoclay	6	-
9	Vorex	80	E-Glass	20	Cloisite	10	-
13	Vorex	80	Kevlar	1	Cloisite	3	-
17	Solarez	60	Kevlar	1	Cloisite	3	-
10	Vorex	20	CF 100	3.7	Cloisite	6.3	-

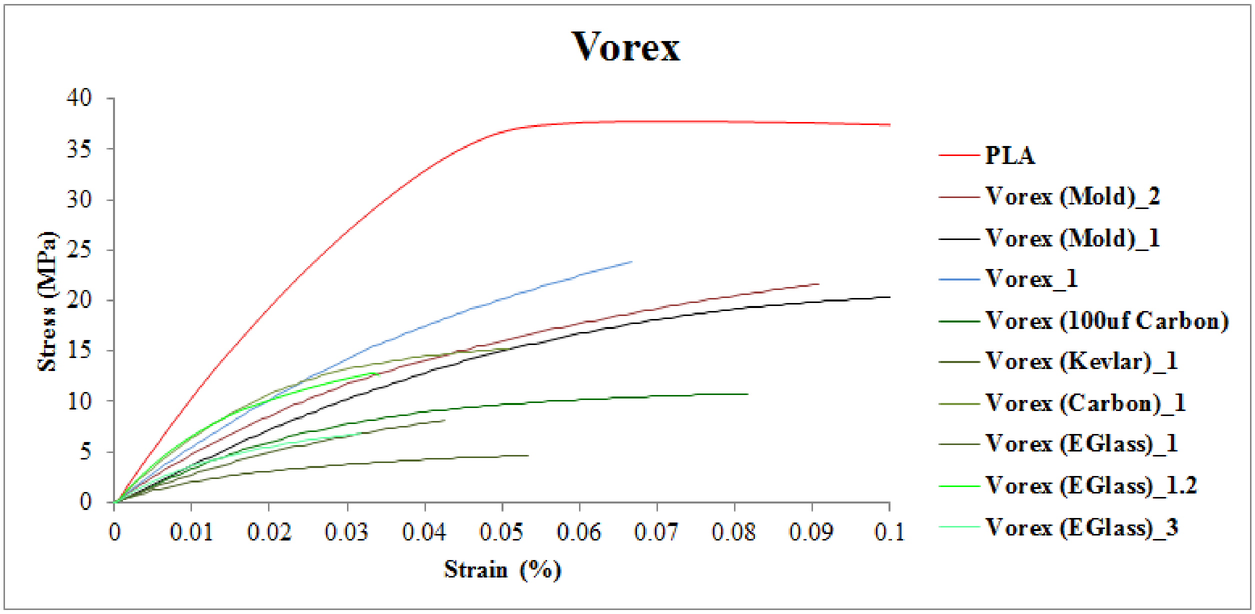

Figure 4: Stress-strain graph of Vorex matrix alone, Vorex/fiber composite of different fiber combinations, and as-printed PLA as a comparison.

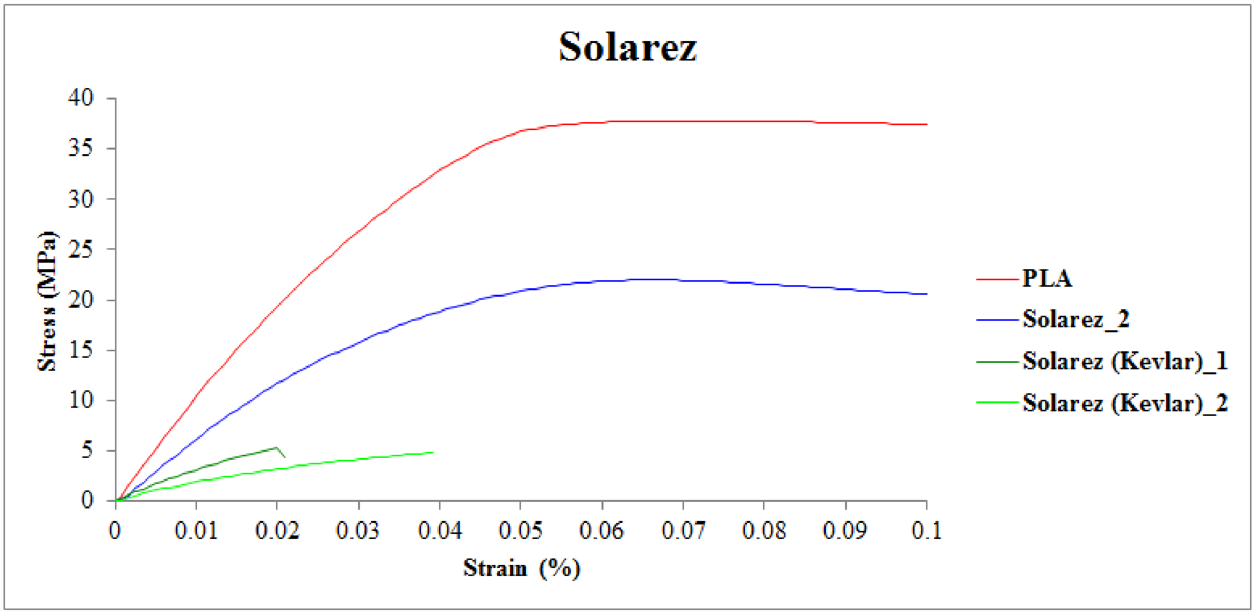

Figure 5: Stress strain graph of Solarez matrix alone, Solarez and different fiber combinations, and as-printed PLA as a comparison.

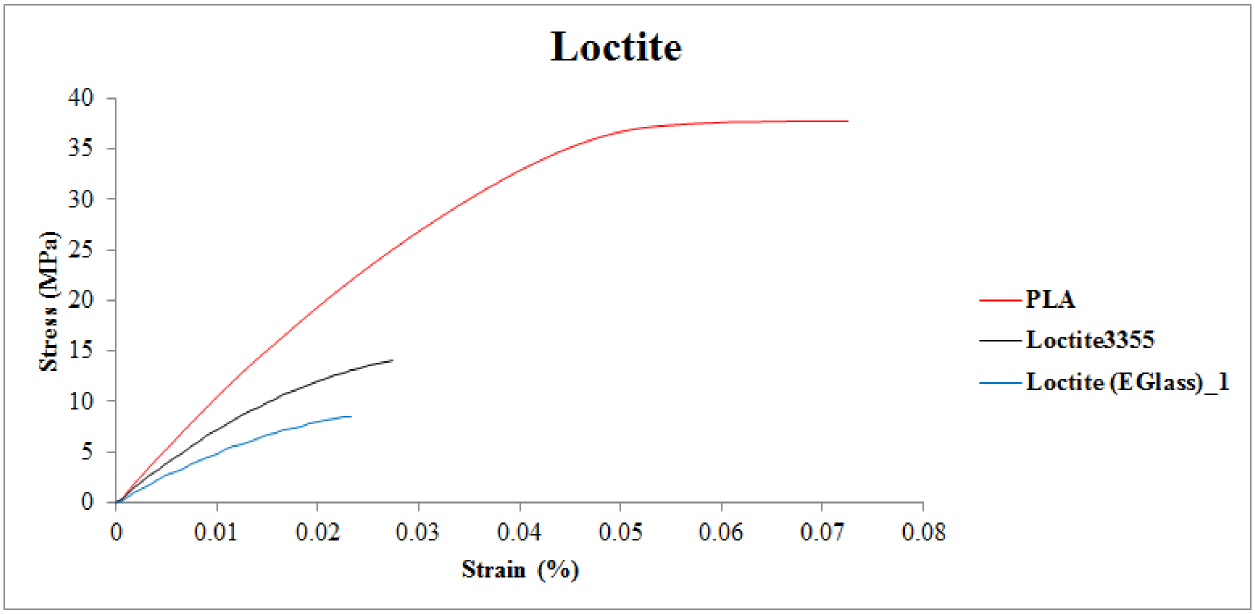

Figure 6: Stress-strain graph of Loctite matrix alone, Loctite with different fiber combinations, and as-printed PLA for comparison purpose.

5.1. Influence of fibers

The fiber type influenced print quality where it was found that the 250 milled carbon fibers were too long producing clumps in the mixture with Vorex and was not able to extrude out of the 14 gauge needle. An attempt with a larger syringe was used and was unsuccessful because the mixture would not extrude and the larger syringe was not able to mount to the modified extruder. The CF100 provided a printable composite with Vorex at low volume fraction. It was found that more than a 10% volume fraction of carbon fibers inhibited the curing time. This was due to the fibers blocking UV light from curing the matrix surrounding the fibers. The carbon nanofibers at less than 2% volume fraction mixed very well and printed easily, but the saturation of these nanofibers caused the matrix to not cure due to blocking of the UV light from curing surrounding matrix. The Kevlar pulp was not easily mixed into each matrix causing a very lumpy mixture and poor print quality if it printed at all. E-glass provided good printing capabilities and faster curing than matrices containing carbon fibers.

5.2. Influence of matrix

Each matrix influenced print quality as well. Vorex cured in one to four seconds when exposed to the UV light. Nevertheless, the extent of curing is also intimately dependent on the amount and type of fibers present in the mixture. Vorex also was easily mixed and would not cure in the syringe or container. Solarez was sensitive to light where it could slightly cure in the container or syringe if the vessel was exposed to sunlight or room light (fluorescent lamps). The samples with Solarez were purposely taped or wrapped to mitigate exposure to light. We observed that while Solarez printed well, the extruded paste did not cure instantly in the presence of UV light. It would gel but not fully cure, indicating the monomers were not completely crosslinked possibly due to insufficient irradiation. Each of the Solarez samples were later cured fully in with prolonged exposure to sunlight.

5.3. Influence of printing parameters

Settings in the slicing software allowed the control of flow and print speed. Each composite mixture had a different consistency where the printer settings had to be adjusted. The flow rate, layer height, and speed affected the time the layers were exposed to the UV light which would affect interlayer bonding. Having a slow speed would produce completely cured layers and a fast speed would provide a partially cured layer. The completely cured layers caused poor print quality when the needle would run over the previous layer that had clumps or air bubbles. These cured hard layers caused the needle to divert from its path and lay more material in those areas causing layer build up and low quality and non-usable samples. In some samples the layers would harden fully preventing subsequent layers from bonding to previous layers.

5.3. Influence of other additives and mixing method

The addition of bentonite clay—an agent that promotes shear-thinning—made the mixture thicker, more consistent, and easier to print. On the other hand, the addition of silicon carbide whiskers made each mixture very thick and difficult to extrude. The silicon carbide also produced bigger agglomerates which would not disperse even when mixing for long times. The presence of air bubbles also inhibited interlayer bonding and weakened the overall sample. These air bubbles are likely due to a suboptimal mixing technique. Additional processing of the composite material is needed to eliminate agglomeration and air bubbles.

6. Insights on student learning

Pre- and post-summer research internship surveys[5] of participating students were conducted to investigate the effectiveness of the research program in reaching its intended goals, namely, to attract and retain minority students into the STEM field. These results are included in Table 2 below, using a 1 (Strongly Disagree) to 5 (Strongly Agree) scale for the pre- and post-internship responses. Comparing pre- and post-summer research internship surveys of participating students, it was found that after the 10-week summer internship, community college students (i) gained stronger understanding of what a STEM career path entailed and how it aligned with their own career aspirations, (ii) were more likely to pursue a STEM career, (iii) had a more informed perspectives on the nature of the work of researchers. For many of these students, the most beneficial aspect of this internship is the imparting of the scientific method of research, which were otherwise not readily understood and learned at a two-year community college, whose focus is on foundational courses. Students reported a deeper understanding of the research process in their field, stronger ability to analyze scientific data and interpret results, keener sense of how theory and practice are integrated, and most importantly, an appreciation of how scientists work on real problems. In addition, because students had an active involvement in knowledge construction rather than being a passive receiver of knowledge, they gained first-hand appreciation of the importance of being resilient as a researcher. Outcomes from this survey support and validate the significance of this collaborative summer internship program between Canada College and San Francisco State University, and underscore the importance of research mentorship as a critical means of attracting and retaining minority students in the STEM field.

Table 2. Pre- and post-summer research internship surveys conducted over 20 participating community students.

Question	Pre-Internship	Post-Internship	Improvement (%)
I understand the research process in my field.	3.21	3.86	20.25
I understand how scientists work on real problems.	3.61	4.28	18.56
This internship clarifies whether I wanted to pursue a STEM research career	3.79	4.36	15.04
I have the ability to integrate theory and practice.	3.61	4.07	12.74
I have skill in interpreting results.	3.86	4.32	11.92
I have the ability to analyze data and other information.	3.96	4.39	10.86
I have a clear career path.	3.79	4.14	9.23
I understand how knowledge is constructed.	3.86	4.21	9.07
I have tolerance for obstacles faced in the research process.	4.04	4.39	8.66
I am ready for more demanding research.	3.82	4.14	8.38
I understand science.	3.71	4.00	7.82
I understand that scientific assertions require supporting evidence.	4.25	4.43	4.24
I am confident I will complete a BS in a STEM field .	4.71	4.89	3.82

7. Conclusions

We have successfully designed and built an extruder system capable of printing short fiber-photopolymer composites on an existing 3D printer platform. The 3D-printed short fiber-infused photopolymer composites was evaluated by visual inspection to determine 3D printing quality and by performing tensile tests on test specimens of standard sizes. We further compared the ultimate tensile strength and elastic modulus of different combinations of fiber/matrix composites against as-printed polylactic acid thermoplastics. Tensile test results showed that the composite samples had lower UTS and elastic modulus than printed PLA samples. Based on observation using a scanning electron microscope, sub-optimally printed composites contained a significant amount of voids between layers attributed to the incomplete adhesion between the layers of prints and air bubbles that were trapped in the photopolymer during mixing. Such defects are likely the results of extensive cavitation during mixing, as well as less-than-optimal print speed, layer height, flow rate, and curing rate. In addition, the lower strength of the short fiber-infused polymer also point to incomplete curing of the inner core of the polymers, due to light shielding by fibers such as carbon and Kevlar. As such, further optimization of the system is required to produce mechanically enhanced short fiber-reinforced photopolymer composites. Lastly, the process of researching, designing, and implementing from thought to fruition is a valuable educational experience for the community college students involved in this project and reinforces their interest in a STEM career. Students reported significant gain in their understanding of the research process, insight into how scientists work on real problems, and overall increase in confidence about their analytical ability.

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