FEASIBILITY STUDY OF EXTRUSION BASED ADDITIVE MANUFACTURING

PROCESS FOR CREATING BIOMIMETIC MICROCHANNEL NETWORK

A Thesis

by

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MASTER OF SCIENCE

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ABSTRACT

Fabrication of micro-scaled 3-dimensional channels to produce a vascular network of capillaries through a sacrificial material approach has been achieved using traditional methods such as casting, lithography, etc. Casting produces a randomly aligned capillary network which provides no predominant direction for flow; this depends on how the sacrificial material settles during the process. Lithography is not known to produce encapsulated channels in a 3D matrix and generally works over the surface or exposed area of the material. Adopting an extrusion-based additive manufacturing method assists the production of aligned capillaries predominantly in the direction of extrusion. This work attempts to characterize the degree of alignment of the fibers of sacrificial material (sugar) in an extruded matrix of PDMS (Sylgard 184™). The goal of this research work is to understand the alignment of sacrificial material fibers in a viscous two-phase extrudable material. Finding an appropriate material combination is under the future scope of this research. Experiments were conducted for identifying a suitable composition of sugar fibers and resin that can be extruded at different pressures to obtain an observable network of fibers under the microscope. Multiple methods of fiber mixing were explored for uniform dispersion and a standard operating procedure for the fiber-silicon extrusion has been proposed. Images of extruded samples for different process parameters were analyzed statistically to quantify the alignment of fibers. A full factorial analysis of experimental design was performed to establish the effect of extrusion pressure and convergence angle at extrusion on the alignment of extruded fibers.

DEDICATION

I dedicate this work to my late grandfather Nana (Dattatraya W. Tare), who always pushed me towards excellence and motivated me to fulfil my dreams.

ACKNOWLEDGEMENTS

I express my gratitude towards my committee chair, Dr. Tai, and my committee members, Dr. Wang, Dr. Li, and Dr. Jarrahbashi, for their guidance and support throughout this research.

I would also like to thank my friends, lab-mates at the Manufacturing Innovation Lab, and the department faculty for making my time at Texas A&M University an unforgettable experience.

The support and encouragement from my mother, my father, and my younger brother during these crucial years of my academic career at grad school has been immense and their contribution cannot be thanked enough. Lastly, I thank the god of wisdom Lord Ganesha for giving me the strength and ability to overcome all the obstacles and achieve my goals.

CONTRIBUTORS

This work was supervised by a thesis committee chaired by Dr. Bruce L. Tai. The committee consisted of Dr. Ying Li, and Dr. Dorrin Jarrahbashi of the Department of Mechanical Engineering; Dr. Jyhwen Wang of the Depart of Manufacturing and Mechanical Engineering Technology.

Experiments were assisted by Mr. Jay Raval, PhD candidate in Mechanical Engineering at Texas A&M University. All the other work included in this thesis was completed by the student independently.

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1. INTRODUCTION

1.1. Motivation

Vascular structures with interconnected capillaries are a common characteristic of tissues in animals as well as plants. A dense network of capillaries carrying blood is present around the lining of lungs in animals which enables the exchange of oxygen and carbon dioxide. A similar network of micro-scaled capillaries is present throughout the volume of human kidneys for the separation of nitrogenous waste products from the blood. In plants, the vascular tissues xylem and phloem are present which carry water and other nutrients from the soil underneath to the farthest leaf at the top. The network observed in plant tissues is different in terms of the alignment of these capillaries from the one that is observed in animal tissues. This is due to the difference in their functions in respective systems. Cross-section images of plant stems show that the capillaries in plant tissues are more aligned to the direction of plant growth which assists the transport in desired direction[1]. Previously, researchers have come up with several different techniques for producing such micro-scaled vascular structures artificially. This includes sacrificial casting, lithography, layer by layer deposition of 2D layers to create desired 3D vascular geometry. Novel methods such as using electric discharge inside a plastic substrate to vaporize material and create branched channels have also been explored. However, all these methods except the layered lithography technique, lead to creation of channels with completely randomized and unpredictable orientation of the channels. Therefore, a method to achieve desired direction of alignment of the microchannels is the next step. Using sacrificial continuous long fibers as the second phase in the two-phase material and extruding it could potentially produce aligned and interconnected microchannels after the sacrificial material have been removed. The hypothesis here is that extrusion assists the degree of fiber alignment which can be controlled by adjusting the extrusion parameters such as extrusion pressure, velocity, and nozzle geometry.

1.2. Literature Review

Creating aligned microchannels artificially is a conundrum which when solved, may unleash multiple facets for future research in the field of biomedical science, biomimicry, and tissue engineering. Extrusion based additive manufacturing has been previously used to deposit two-phase viscous materials which contain micro-sized chopped fibers [2,3]. Attempts to print continuous fibers have also been made with multiple nozzles or separate deposition of fibers and base material [4]. Assisting extrusion with acoustic energy to align fibers produced promising results for short length reinforcements of sub-millimeter lengths [3]. Artificially fabricated structures which mimic microvascular channels have great value in biomedical research. A layer-by-layer approach with 2D fabricated layers using standard microfabrication techniques such as photolithography has been implemented before to create 3D structures with micro-sized channels [5]. This allows for organized and systematic positioning of the channels, but the layer assembly poses issues with precision and interconnectivity of channels in the build direction. A branched network of capillaries can also be produced inside plastic material using electric discharge [6]. This method uses high energy density discharge of electrons which locally vaporizes material to create fractal-like networks inside the plastic matrix.

Another popular approach for creating micro-scaled channels or features, in general, is to use a sacrificial material in the matrix of the base material. This method is widely used to create open-celled foams with controlled pore size [7]. The use of sacrificial materials to fabricate intricate features inside 3D printed parts has been explored for various applications [8,9,11]. Creating new sacrificial materials by modifying extrudable polymers such as PLA(poly-lactic acid) with catalytic nanoparticles is another way of achieving desired features by material removal [11]. Sugar fibers have been used as a sacrificial material in the casting process to produce channels with a diameter of the order of 10µm [10]. Moving iron loaded magnetic fibers from casted gelatin hydrogels to create desired topology of 3D channels proves to be effective for microfluidic applications and preparation of micro-valves [12]. This technique, although unique, requires preparation of special magnetic fibers and electromagnets for channel opening.

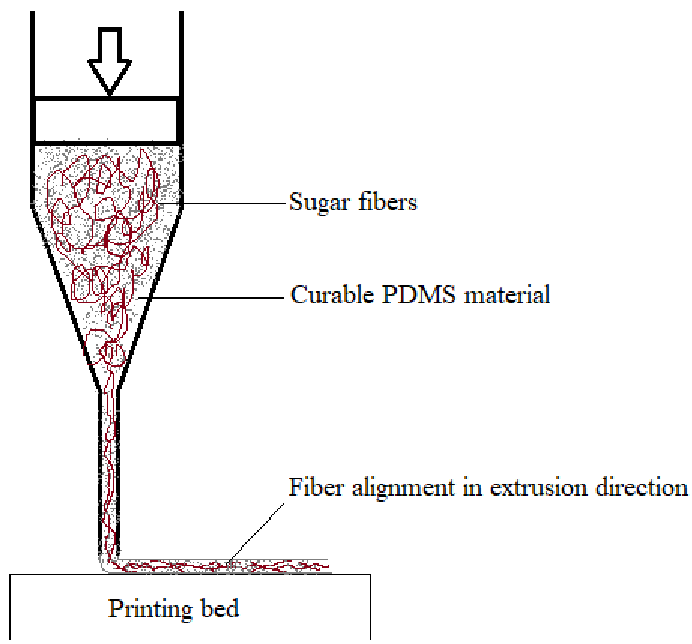

Figure 1.1 Extrusion of sugar fiber + PDMS material leading to alignment in the printing direction

A wide range of applications such as filtration, energy storage, reaction catalysis and even biomedical devices have found porous materials quite useful owing to their versatile nature. Traditionally, micro, and nano sized porous membranes have been fabricated using the principle of phase inversion. More recently, the ability to 3D print geometrically complex shapes of polymers has opened an entirely new chapter in manufacturing of these porous structures. Using suspended nanoparticles as sacrificial materials, emulsified inks with sacrificial templets are some of the commonly used methods to produce porous structures via 3D printing; they require post processing that involves removal of sacrificial material from the printed matrix. A similar approach was used by Alison et. al. [13] for this purpose. Emulsion-based inks were developed for additively fabricating hierarchically porous material with pore size ranging from few hundred nanometers to few millimeters. Removal of nanodroplets and micro scaled templets in the initial ink gives open-celled porous structure. Industry has developed commercial solutions for printing porous materials using sacrificial approach. FDM filaments such as PORO-LAY contain sacrificial material that is removed after printing. Most of these techniques involve post processing for formation of pores. To eliminate post processing, Karyappa et. al. demonstrated a novel method termed as ip3DP [14]; where the polymeric inks were printed directly in a non-solvent bath and *in situ* immersion precipitation was achieved.

Chen et.al. [15] successfully printed a highly elastic and durable foam with sacrificial approach using Direct Inkjet Writing (DIW) technique followed by acid etching of sacrificial material and phase inversion. Going a step ahead, they introduced CNT in their ink and obtained a piezoelectric effect using same fabrication method. Most of the recent research in the field of 3D printing of porous materials, shows development of emulsion-based inks with sacrificial material and optimization of their composition to obtain printable material rheology. A more advanced approach for fabrication of membranes with aligned pores using self-aligning particles was perceived by Yang et.al.[16]. This involved using nanodomains of homopolymer PMMA in its copolymer as sacrificial material. The cylindrical nanodomains align themselves in the direction normal to surface and when etched with HF, a thin film with aligned pores is obtained. This showed that self-aligning particles can produce directionally dominant porous materials. In past, researchers worked their way around with fiber reinforcement in polymer filaments. Barbosa et.al. [17] attempted to establish a correlation between fiber concentration and fiber-fiber interactions which ultimately affects the fiber orientation in the extruded polymer loaded with fibers. However, this effort was limited to short length fibers and not sacrificial in nature. Continuous fiber printing has also been explored in the past for understanding improvement in the material strength [19]. Feasibility of printing continuous long fibers was affirmed by Chatterjee et. al. in their work to produce textiles via extrusion-based 3D printing [20]. It is evident that fiber printing has been used for various applications and there has been a little effort to explore feasibility of sacrificial printing of long continuous fibers.

1.3. Scope of study and hypothesis

As discussed before, the existing methods of fabricating micro-channels do not provide enough control on the alignment direction. During the process of extrusion of viscous material with sacrificial fibers as secondary phase, a shear profile is developed across the cross section of extruder. An experimental study done by Peng et.al. [18] explained this phenomenon for a single-phase polymer material in additive manufacturing process that uses melt extrusion. Assuming that the flow during extrusion is primarily governed by the base phase of the mixture, this flow profile should exert a drag force on the fibers and force them to align with the extrusion direction or the material flow direction during the extrusion process. Every extrusion parameter that induces higher shear rate in the flow, assists alignment. Shear rate in a flow is defined as the rate of change of velocity in the direction perpendicular to the direction of flow. Intuitively, one can say that higher the difference in velocity of flow in consecutive layers, higher is the shear force. The fibers will try to align themselves in such a way that they experience minimum drag which is the direction of flow of material – extrusion direction. The scope of this study is to understand the degree of alignment of sacrificial sugar fibers within a PDMS base material through the process of extrusion.

The process of extrusion of two-phase mixture with viscous base (phase-I) and sugar fibers (phase-II) is governed by multiple variables including environmental conditions, nozzle geometry, fiber proportion in the material mixture, etc. An experimental design for extrusion has been evaluated in this study to understand the effects of various parameters on the alignment after extrusion. The measured response in this experimental analysis is the angle made by each individual fiber with a datum fiber as observed from the top surface after extrusion. Latter part of the analysis includes statistical parameters such as the mean and standard deviation of the measured angles. The factors considered for experimental design are extrusion pressure (P) and the angle of convergence (C) of the extrusion syringe. Assuming that the flow is governed by the primary phase (viscous base), higher extrusion pressure in the same geometrical conditions of extrusion is assumed to induce higher flow velocities. This was confirmed during the extrusion pressure and velocity calibration in the experimentation stage of this work. Therefore, the primary hypothesis of this experimental work is that a higher shear rate in the flow should lead to higher degree of alignment in the extruded sample. Smaller convergence angle for extrusion between the same initial and final diameters of cross sections will effectively provide more time for the fibers to align with the direction – hence the fiber angles will be closer to the extrusion direction.

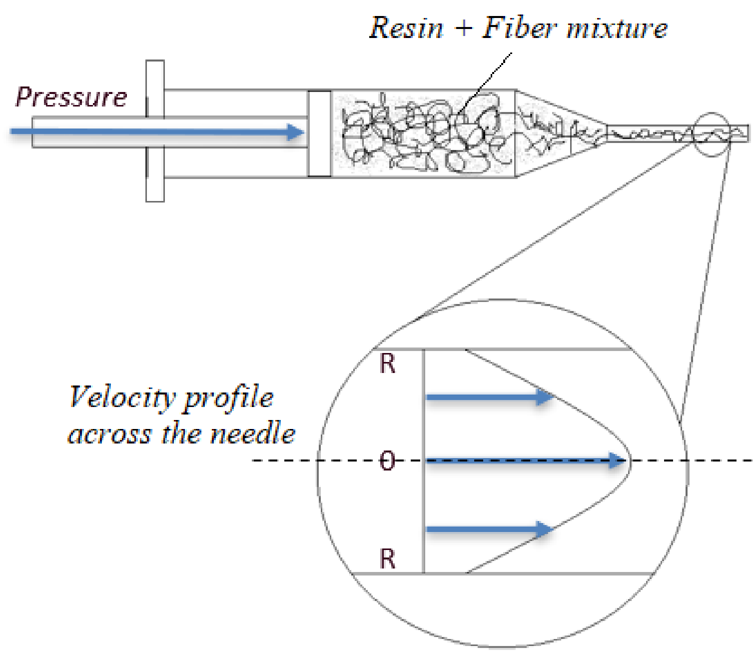

Figure 1.2 Velocity profile of flow inside needle during extrusion of viscous material with fibers

2. MATERIALS, EXPERIMENTAL SETUP AND METHOD

2.1. Materials

For observing embedded fibers or produced channels in extruded samples, clear resins are chosen over other semi-transparent or opaque curable resins. A variety of resins were tried as a base material. Encapso™ K by Smooth-on was found to be non-extrudable due to its low viscosity and high curing time. Dragon Skin™ by Smooth-on was also low viscosity candidate despite being quickly curable. Sylgard-184 by DOW Inc. A clear PDMS based resin - Sylgard 184 by DOW industries – was used by Bellan et. al [10] with sugar fibers to create a vascular network. Sylgard-184 when mixed with sugar fibers, gave the best results for extrusion and was chosen as the base material for further experiments. As mentioned earlier, edible granulated sugar was used in a cotton candy machine to make sugar fibers. These two components were mixed in a fixed proportion to obtain a suitable viscosity mixture for extrusion. Following sub-sections detail out the process of material preparation and mixing.

2.1.1. Resin mixing

This is the first step in the process of material preparation for the experiments. If sugar fibers (cotton candy) are prepared before mixing the resin material, they absorb moisture in the ambient surrounding to form agglomerates leaving no fibers by the time resin is ready for mixing. Sylgard-184 needs to be mixed in a proportion of 10:1 for base to hardener (weight to weight or volume to volume ratio) as per the mixing instructions provided by the manufacturer. Wooden tongue depressors were used to mix resin thoroughly in plastic cup. During the resin mixing process, a lot of air bubbles are trapped inside. These are removed using a vacuum chamber. The prepared resin in subjected to a vacuum of 25mmHg for 30 minutes. This process is commonly referred to as deaeration of the resin.

2.1.2. Sugar fibers preparation

A countertop cotton candy maker was used to prepare sugar fibers. The spinneret of sugar candy machine is allowed to gain temperature before a scoop of granulated white sugar was poured inside it. In the process of fiber formation, high temperature causes the sugar granules to melt. Molten sugar is centrifugally thrown out of the spinneret from the slots on the periphery. As soon as the molten sugar encounters surrounding cold air, it solidifies to form the fibers. These fibers ate then collected using a wooden stick in a direction perpendicular to the spinneret axis as shown in Figure 2.1. It was made sure during the experiments that the sugar fibers are collected in the exact same fashion during all the material preparations. The collected fibers were then transferred to a plastic cup and weighed to required value for mixing with the resin. If the sugar fibers are prepared in advance, they absorb ambient moisture and form sugar globules. So, it is important that the sugar fibers are prepared fresh as the resin deaeration is almost complete.

Figure 2.1 Preparation and collection of sugar fibers using a countertop cotton candy maker

2.1.3. Mixing resin and sugar fibers

This is one of the critical steps of material preparation. Different mixing approaches were explored before finalizing the manual mixing approach for the final experiments. In the initial approach, the resin was allowed to settle through fibers placed inside syringe under gravity. In this method, one needs to pour more resin than required for the mixture composition. This process of settlement under gravity takes a very long time (about 45 to 75 mins) mainly because of high surface tension of the resin and change of viscosity of the resin with time because of the catalyst (hardener) action. Also, as the resin flows down, it pushes the fibers to the bottom of the syringe because of its weight (see Figure 2.2(a)). As a result, the obtained mixture of fibers and resin is not volumetrically consistent in terms of the presence of fibers in the bulk.

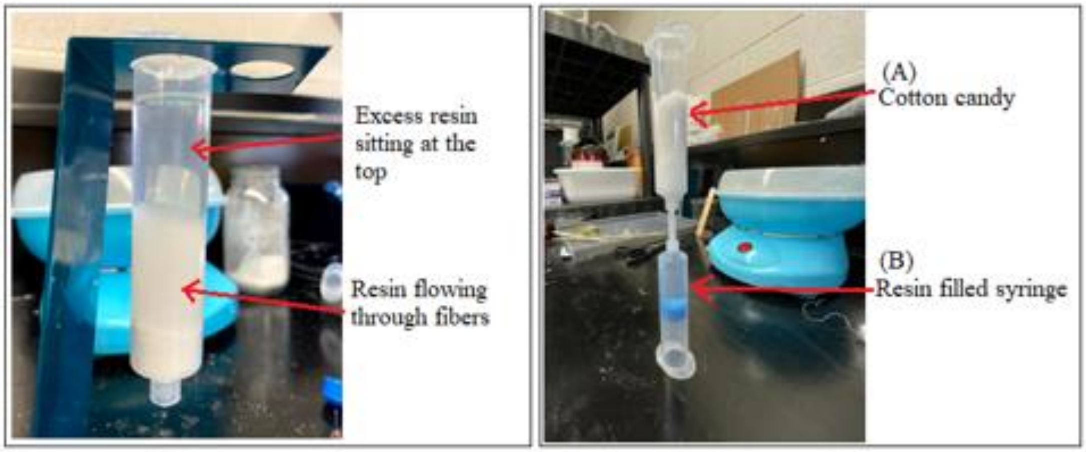

Figure 2.2 Passive mixing techniques (a) gravity mixing (b) two syringe setup for pressure mixing

The second approach was to force resin through fibers using air pressure. In this method of mixing, two syringes were used as shown in Figure 2.2(b). The bottom syringe was filled with resin and top one with the cotton candy. In forced pressure method, the air pressure was applied to the bottom syringe and the resin was forced into the top one through the cotton candy fibers. In this technique, the sugar fibers were lifted by the normal reaction from resin instead of the resin mixing into them. With the same setup, instead of positive pressure, a vacuum pressure was applied to the top syringe so that it sucks resin from the lower syringe. This gave the same type of results in terms of fiber dispersion in the matrix. Finally, a manual mixing approach was decided upon as the most effective one. In this method, the measured quantity of sugar fibers was collected in a plastic cup. Resin was poured on these fibers from the top. After this, a tongue depressor was used to mix the resin over fibers manually. The rate of mixing was maintained at 2Hz. The direction of rotation was maintained either clockwise or counterclockwise throughout the process of mixing. This is to minimize the risk of breaking fibers. With several experiments, it was concluded that the mixing process requires a duration of 90 seconds to obtain a consistent mixture without breaking a lot of fiber strands.

The composition of mixture used for experimental analysis was kept fixed to 1.5 grams of sugar fibers per 20 grams of resin. Assuming standard sugar bulk density of 0.85g/cc, the volume fraction of sugar fibers in the mixture was set close to 9%.

2.2. Extrusion

Standard 30cc plastic syringes with plastic plungers were chosen as the extrusion tool for the purpose of experiments. These syringes have luer lock for attaching compatible needles. The traverse velocity across printing bed needs to be equal to the velocity of material being extruded at any pressure. If the extrusion velocity is less than the traverse velocity, the material leaves traces and does not give a seamless print (case C in Figure 2.3). If the extrusion velocity is more compared to the traverse velocity, the material deposition is more at one location forming blobs on the print bed (case B in Figure 2.3).

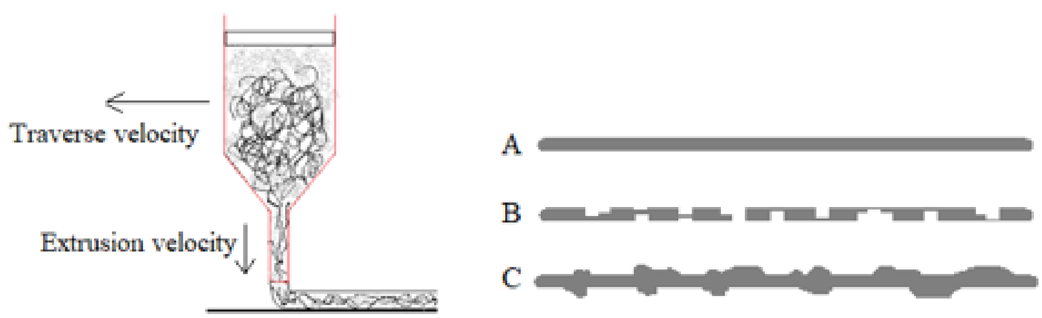

Figure 2.3 Traverse velocity and extrusion velocity

Ideally, the extrusion velocity should be equal to or slightly less than the traverse velocity as a rule of thumb. Through several trial-and-error instances, the printhead of

Flashforge Dreamer 3D printer was calibrated for different extrusion pressures for the set mixture composition. The printhead of Flashforge Dreamer was controlled through open software FlashPrint by the same manufacturer.

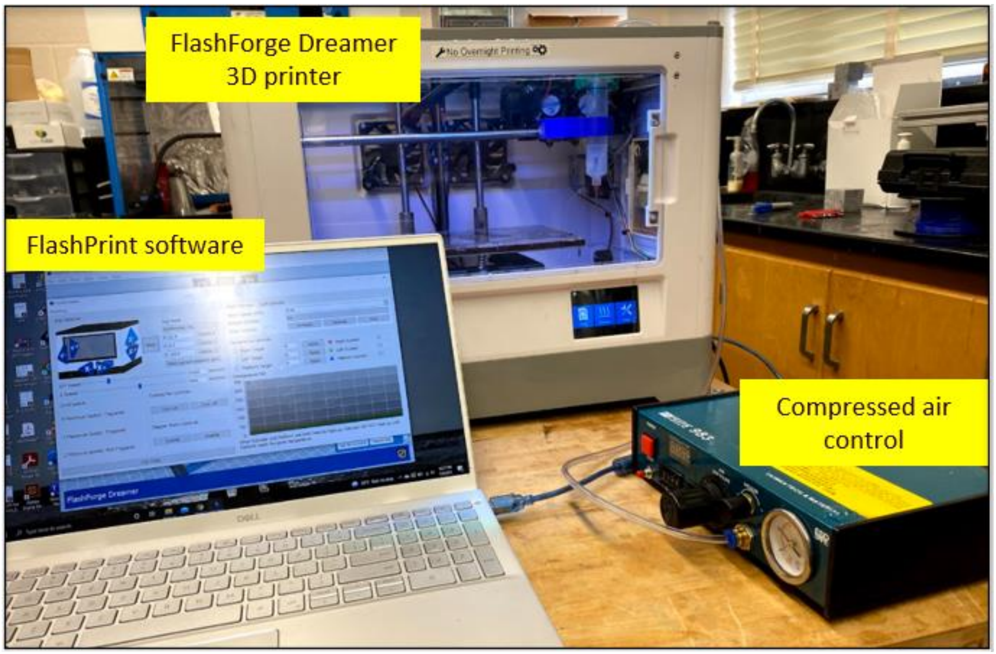

Figure 2.4 Extrusion setup

Following plot in Figure 2.5 shows the calibration curve. When the extrusion pressure is low, the print velocity is inconsistent; on the other hand, for higher extrusion pressures (>40 psi), the material tends to blurt out of the needle, and it is impractical to synchronize high speed traverse with extrusion without a sophisticated control mechanism. Thus, for the scope of this work, a working range of pressures was selected (10 psi $\leq$ P $\leq$ 30 psi).

The extrusion pressure control was done through air pressure regulator with a pressure gage for monitoring. The default convergence half angle of the syringe was measured to be 60°. For achieving smaller convergence angle, nozzle plugs with different half cone angles were designed and printed out on an SLA 3D printer. This additional plug was designed to sit at the bottom of 60° cone angle as shown in the Figure 2.6.

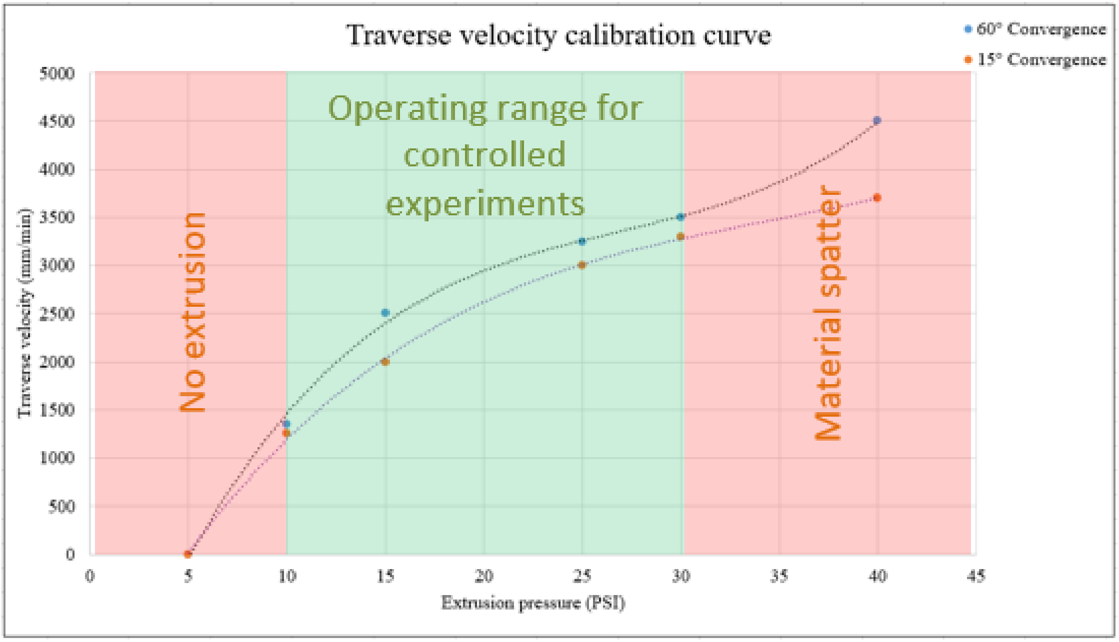

Figure 2.5 Calibration curve for traverse velocity

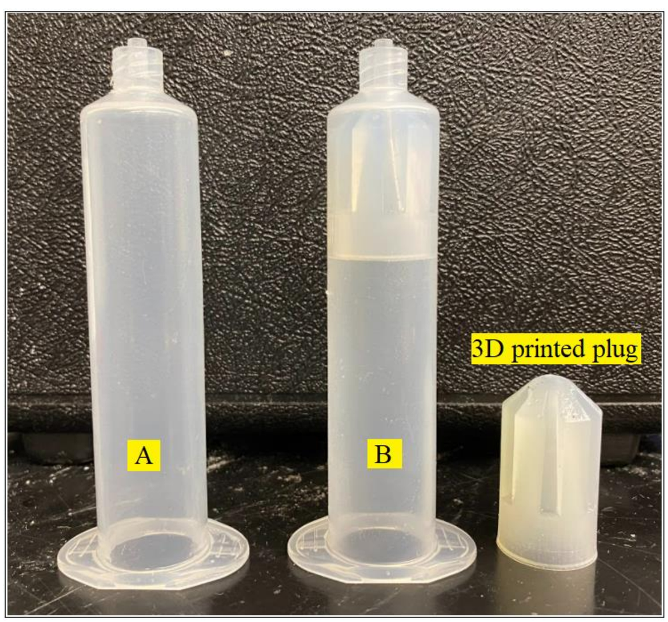

Figure 2.6 3D printed plug for altering the convergence angle for extrusion

2.3. Measuring fiber angles and alignment

The printed samples were observed under microscope camera MU-500. The camera magnification settings were kept consistent throughout all the image captures. A square shaped area of 2mm by 2mm is captured. Each of the images was marked and analyzed using Solidworks™ software. Image was captured such as the printed sample aligns with capturing camera horizontally as shown in Figure 2.7. In each sample image, 51 fibers were marked using the sketch tool and one of the fibers was fixed as the datum for analysis of that image sample. Angle between all the fibers with the datum fiber was measured using the measure tool. This gave a total of 50 data points per image. These values were captured with a sign convention as shown in Figure 2.8. The absolute value of all the angles was captured between 0 to 90 degrees.

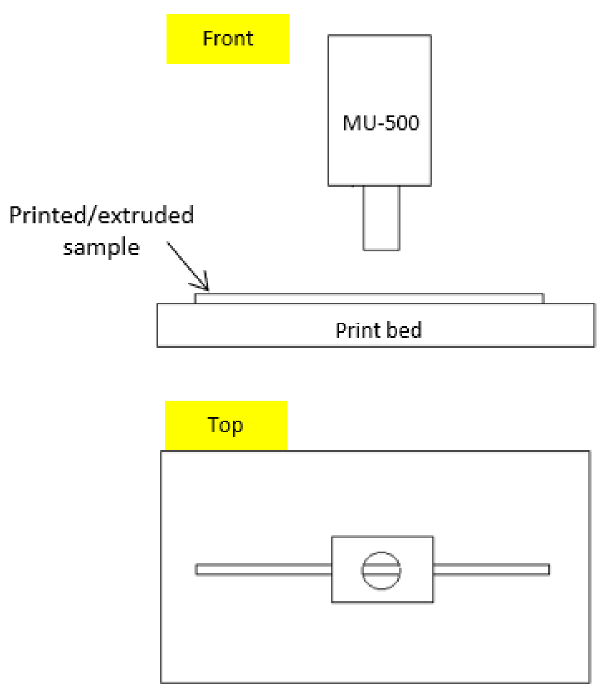

Figure 2.7 Image capture orientation

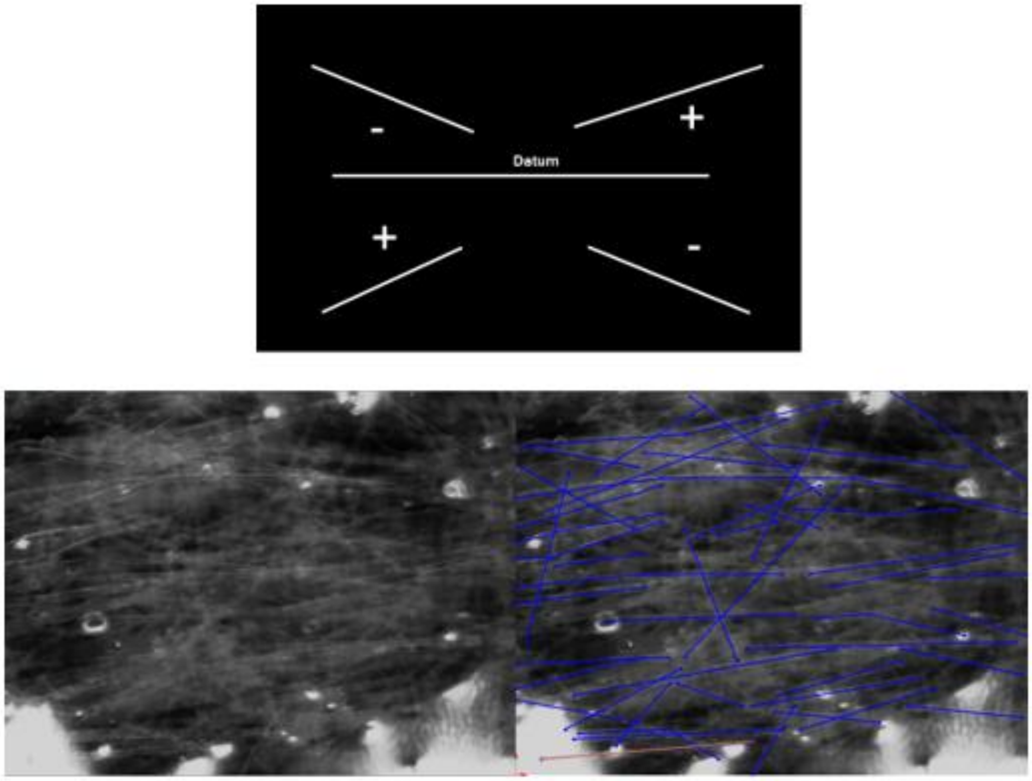

Figure 2.8 Angle measurement convention and sample angle marking for one image

3. EXPERIMENTAL DESIGN

3.1. Full factorial design

For the experimental design, extrusion pressure and angle of convergence of the syringe were considered as the control parameters. The pressure (P) was varied in 3 steps 10PSI, 15PSI and 30PSI. For the convergence angle (C), two levels were chosen for full factorial analysis viz. 15° and 60° half angles. Thus, it is a 3x2 full factorial design with total six experiments. This design of experiments had three repetitions for the test of repeatability as well as improving the credibility of results obtained through the analysis. The other variables associated with the process of extrusion were maintained constant or unchanged for all the experiments. Table below summarizes the experimental design.

Variable	Levels
Extrusion pressure (P)	10PSI, 15PSI, 30PSI
Angle of Convergence (C)	15°, 60°
Needle diameter (D)	14-gage(1.6mm)
Needle length (L)	Constant = 25.4 mm
Material composition	Fixed
Environmental conditions	Not controlled/ambient

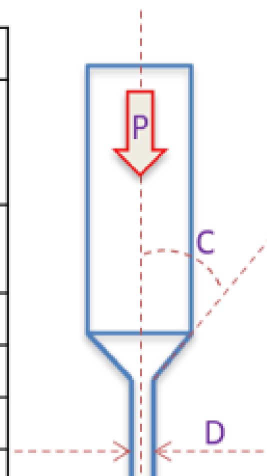

Figure 3.1 Experimental design and variables

Intuitively, the arithmetic "average" of all the recorded angles for one of the sample images seems to be a good response parameter for factorial effects analysis. However, it is observed that the mean of all fiber angles in a single image is always around in the range of [-10°,10°]. This means that all the fibers are aligned in the printing direction or the extrusion direction. But it is important to understand that even if the mean is close to 0, it does not necessarily mean that the fibers are equally aligned in all the experimental conditions. The spread of angle measurements is an important indicator of the absolute alignment of these fibers in the bulk. Figure 3.2 shows how fibers with average deviating away from 0 still have good alignment owing to their spread from the mean value of the dataset.

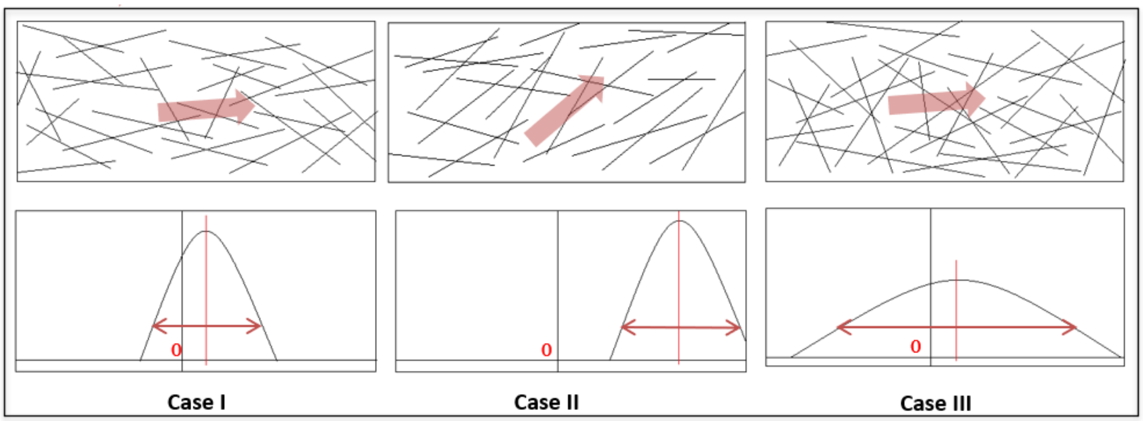

Figure 3.2 Misleading nature of mean of the dataset

If only the mean of all three cases shown in Figure 3.2 is considered, one may conclude that case I and case III are well aligned fiber layouts. But it happens so that the case II fiber layout has more compact spread of the fibers – be it around a value farther away from 0. The preliminary observations about mean of the angles in one image also show that the averages for all of them are almost identical and there is no significant difference in the alignment if only averages are considered for alignment assessment. Thus, the standard deviation of the measured angles needs to be the indicator of how well aligned the sample fibers are (for the recorded values with sign convention). The sign convention has been followed to understand if the data distribution is normal which allows us to analyze the ‘spread’ of data about the mean of the distribution. The mean of each individual dataset could in fact point towards the actual printing direction of that sample. The observed deviation in the mean from 0 could be a resultant of the sample not being perfectly aligned to the camera during the capture of image. As shown in Figure 2.7, the orientation of image capture should minimize the relative angle of printing direction and horizontal of the actual image – which allows us to perform the absolute value average analysis (absolute values of angles without following the sign convention).

3.2. Preliminary observations

After performing first set of 6 experiments, the images were marked, and alignment of fibers was observed. In the first impression from a visual inspection of the images shows that alignment for lower extrusion pressure is found to be better as compared to the higher-pressure extruded sample. A similar trend is observed with respect to the angle of convergence. The lower convergence angle is found to visibly show a better alignment of the fibers compared to the higher convergence angle. Frequency distribution plots for all these samples also show that the 10PSI, 15° convergence angle sample has the most fibers aligned to the printing direction compared to the other samples (Figure 4.1). Although any further visual comparisons are not conclusive, one can say that low extrusion pressure with low angle of convergence proves to be better for alignment.

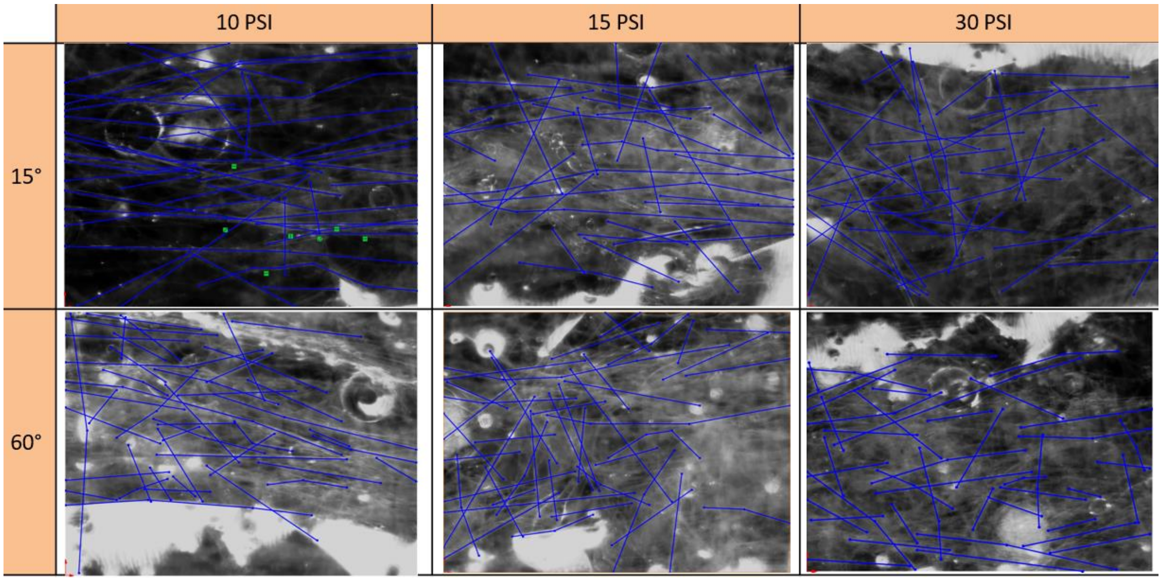

Figure 3.3 Preliminary comparison of all 6 printing condition samples

4. RESULTS AND DISCUSSION

4.1. Mean of the captured angles

Figure 4.1 shows the boxplot distributions of all the 6 experiments each with their 3 repetitions together. The legend follows a nomenclature (Pressure-Convergence). Preliminary visual impression for this plot suggests that all the means are almost equal to 0 and all the printing conditions produce similar alignment (assuming this mean with signed values of angles is the right metric for assessment).

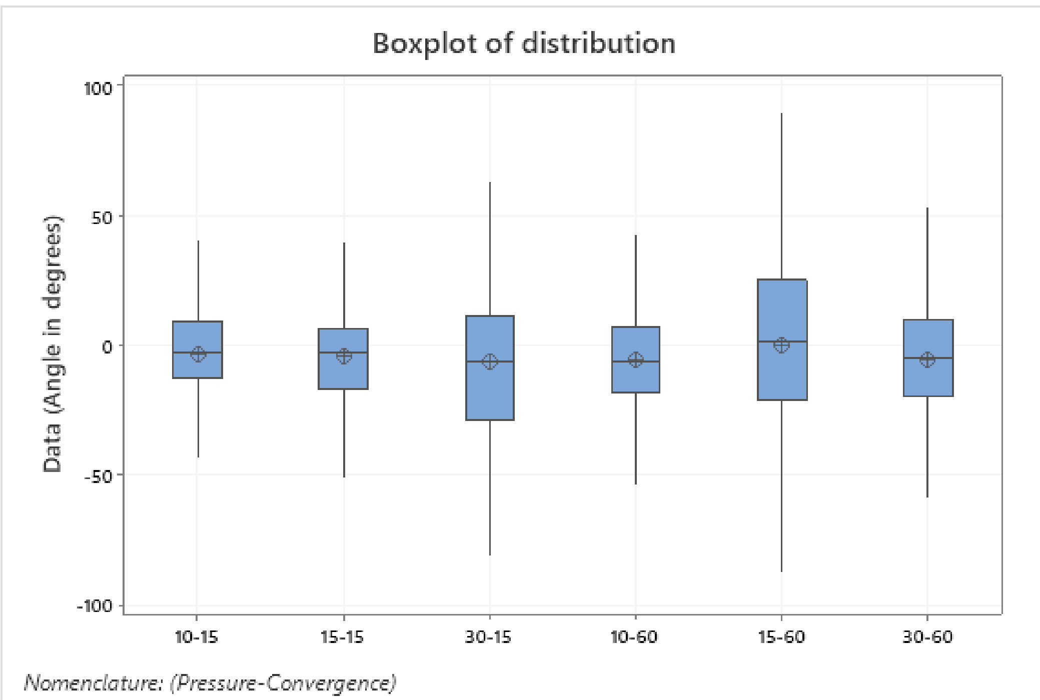

Figure 4.1 Boxplots of angle measurements

To verify the statistical significance of the difference in the means of all these different datasets, a one-way ANOVA was performed using Minitab. The results of this analysis suggest that none of the 6 means from the collected data for 6 different experimental settings is statistically different from the group.

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Factor	5	3876	775.3	0.83	0.526
Error	896	833530	930.3		
Total	901	837407			

Method

Null hypothesis	All means are equal
Alternative hypothesis	Not all means are equal
Significance level	α = 0.05

Equal variances were assumed for the analysis.

Grouping Information Using the Tukey Method and 95% Confidence

Factor	N	Mean	Grouping
15-60	149	-0.17	A
10-15	155	-3.28	A
15-15	159	-4.44	A
30-60	146	-5.57	A
10-60	151	-5.71	A
30-15	142	-6.43	A

Means that do not share a letter are significantly different.

Figure 4.2 One-Way ANOVA on all 6 datasets of angle measurements

Thus, one can conclude that mean of the data with sign convention is not a good concluding indicator for understanding the effects of operating parameters. The Tukey test of HSD (Honestly Significant Difference) shows that all the datasets fall in the same group with 95% confidence level. However, considering positive and negative values of angles from datum is same as considering negative value for troughs and positive values for crests for a surface roughness measurement. This is likely to suggest that the surface is smoother than it is. So, only the absolute values of these angles were considered for the analysis of means. Figure 4.3 below shows the extreme value distribution fits for the absolute values of angles measured with respect to the datum fiber. Interval plots with mean at 95%

confidence level suggests that the extrusions at 10 PSI tend to be more effective for alignment in the direction of extrusion.

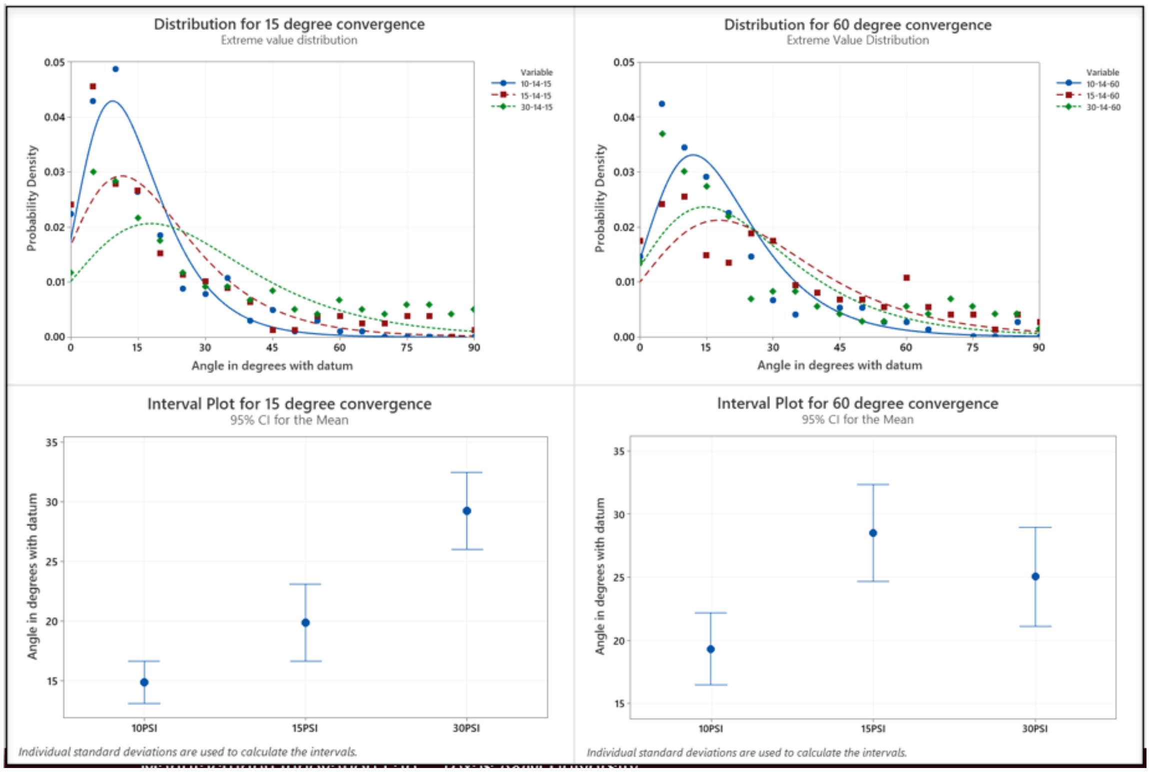

Figure 4.3 Extreme value distribution fits and interval plots of absolute value data

A factorial analysis was run on this parameter – the ‘mean’ of combined data with absolute values of angles as the response (Figure 4.4). For both control factors, the p-value calculated via ANOVA is less than 0.05 for 95% confidence; hence, we conclude that both pressure and convergence angle of the extrusion setup affect the degree of alignment. The effects plot (Figure 4.5) show that low pressure extrusions produce better alignment or lower average angle with datum. Later, the analysis of standard deviation as a response, also shows similar trend in the effect which confirms these results.

Factor Information

Factor	Levels	Values
Pressure	3	10 psi, 15 psi, 30 psi
Angle of Convergence	2	15 deg, 60 deg

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Model	5	31588	6317.6	14.04	0.000
Linear	3	20746	6915.4	15.37	0.000
Pressure	2	18701	9350.3	20.78	0.000
Angle of Convergence	1	2254	2254.2	5.01	0.025
2-Way Interactions	2	7299	3649.7	8.11	0.000
Pressure*Angle of Convergence	2	7299	3649.7	8.11	0.000
Error	1043	469314	450.0		
Total	1048	500902			

Figure 4.4 Factorial analysis ANOVA on absolute values of angles made by fibers with respective datum

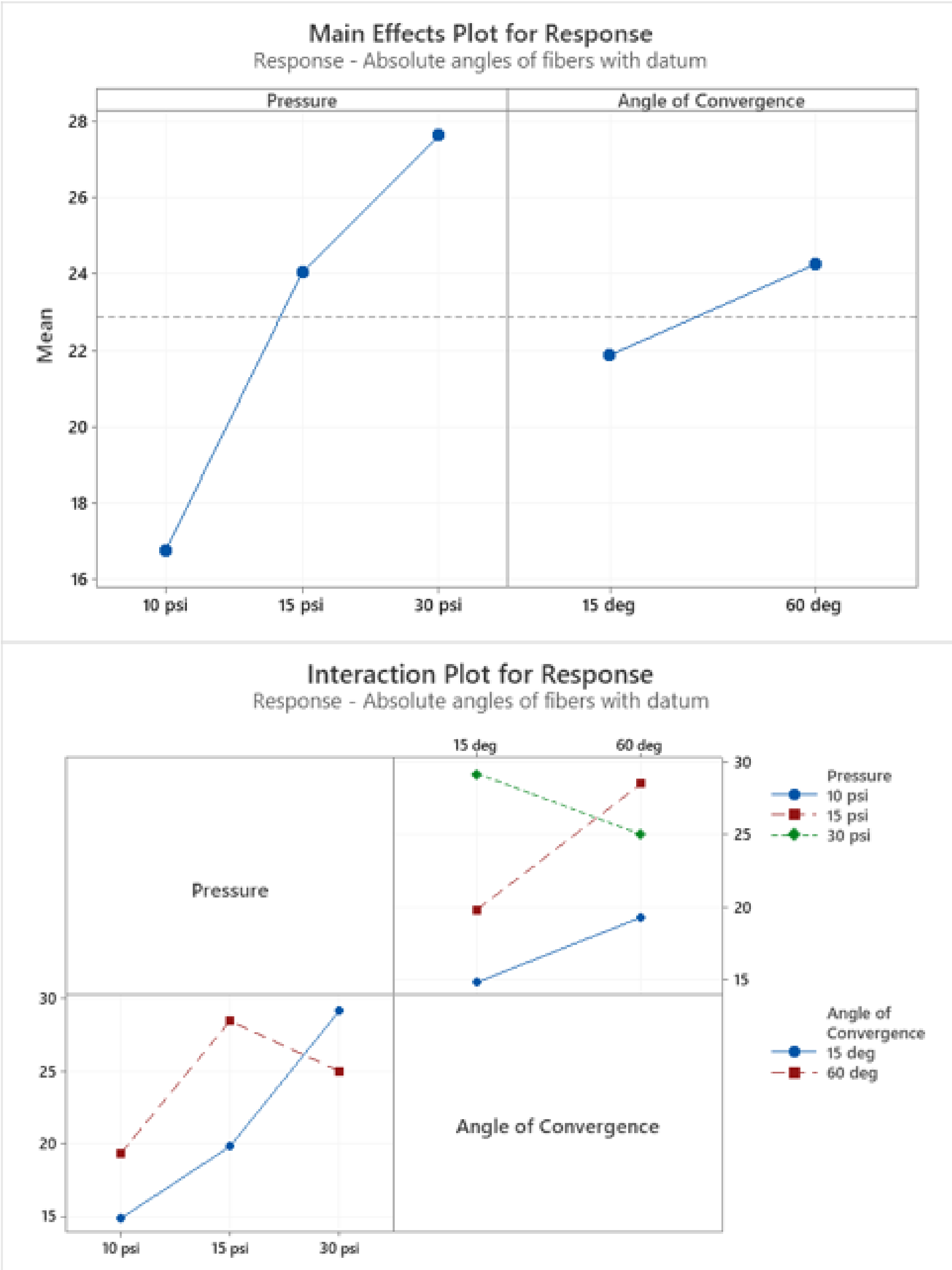

Figure 4.5 Effects plot for absolute angles as response

4.2. Spread of the data – Standard Deviation

Figure 4.4 shows visually that even though the means are indistinguishable, the spread of measured angle values with sign convention about the respective means is significantly different in all the cases. This is something we are interested to find out; since more compact spread indicates better alignment and that all the fibers are pointing in a particular direction. Larger spread of the data indicates randomness in the data and shows that the alignment is not consistent. Applying this analogy, another analysis on the standard deviations of the individual repetitions in the design of experiments was performed. The standard deviation for each replica of experiment was considered as the response for a full-factorial analysis.

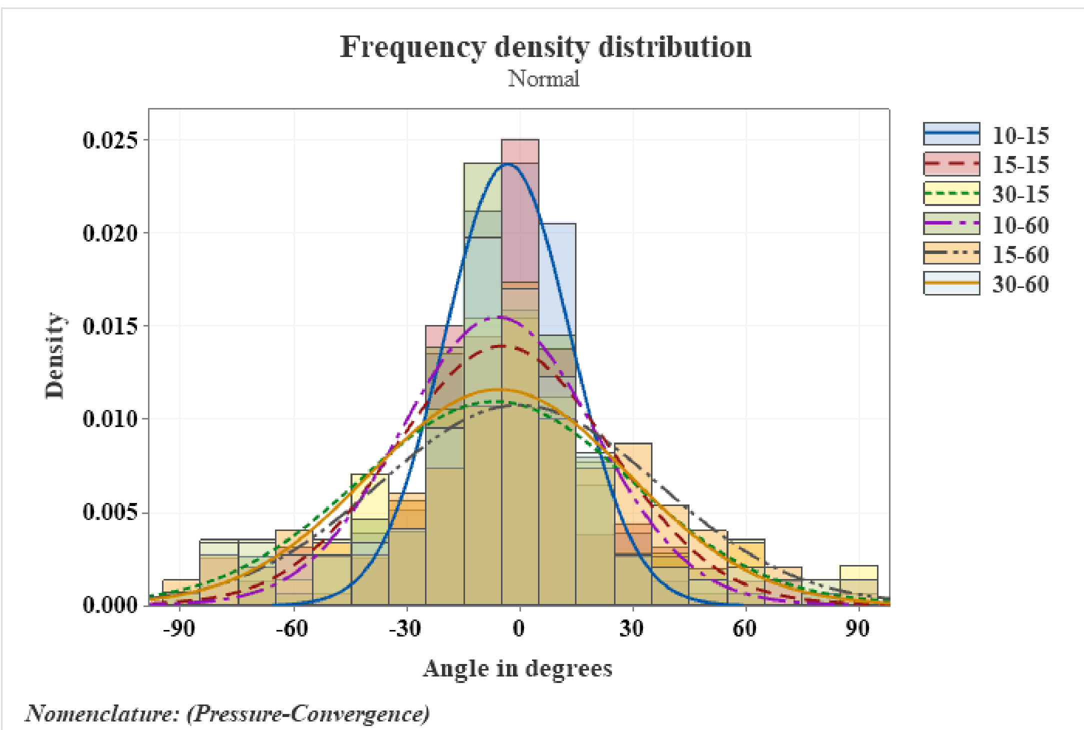

Figure 4.6 Frequency density distribution

4.3. Factorial analysis for standard deviation as a response

As discussed before, standard deviation for each repetition of each experiment was considered for factorial analysis to understand the effect of control parameters on the alignment of fibers. Lower standard deviation indicates higher degree of alignment and vice versa. The individual effects plot for this factorial analysis showed that higher pressure extrusions lead to higher standard deviation of the measured fiber angles. For the angle of convergence, the standard deviation of measured angles for lower angle showed a lower value indicating that the low angle of convergence is beneficial for the alignment. For the scope of this work, alignment for the 10PSI extrusion with 15° convergence angle shows best results.

The interaction plots indicate that at higher values, the extrusion pressure does not have the same effect as it has at lower values with a higher angle of convergence. This indicates that if the angle of convergence is high, pressure alone cannot be a factor to predict the degree of alignment.

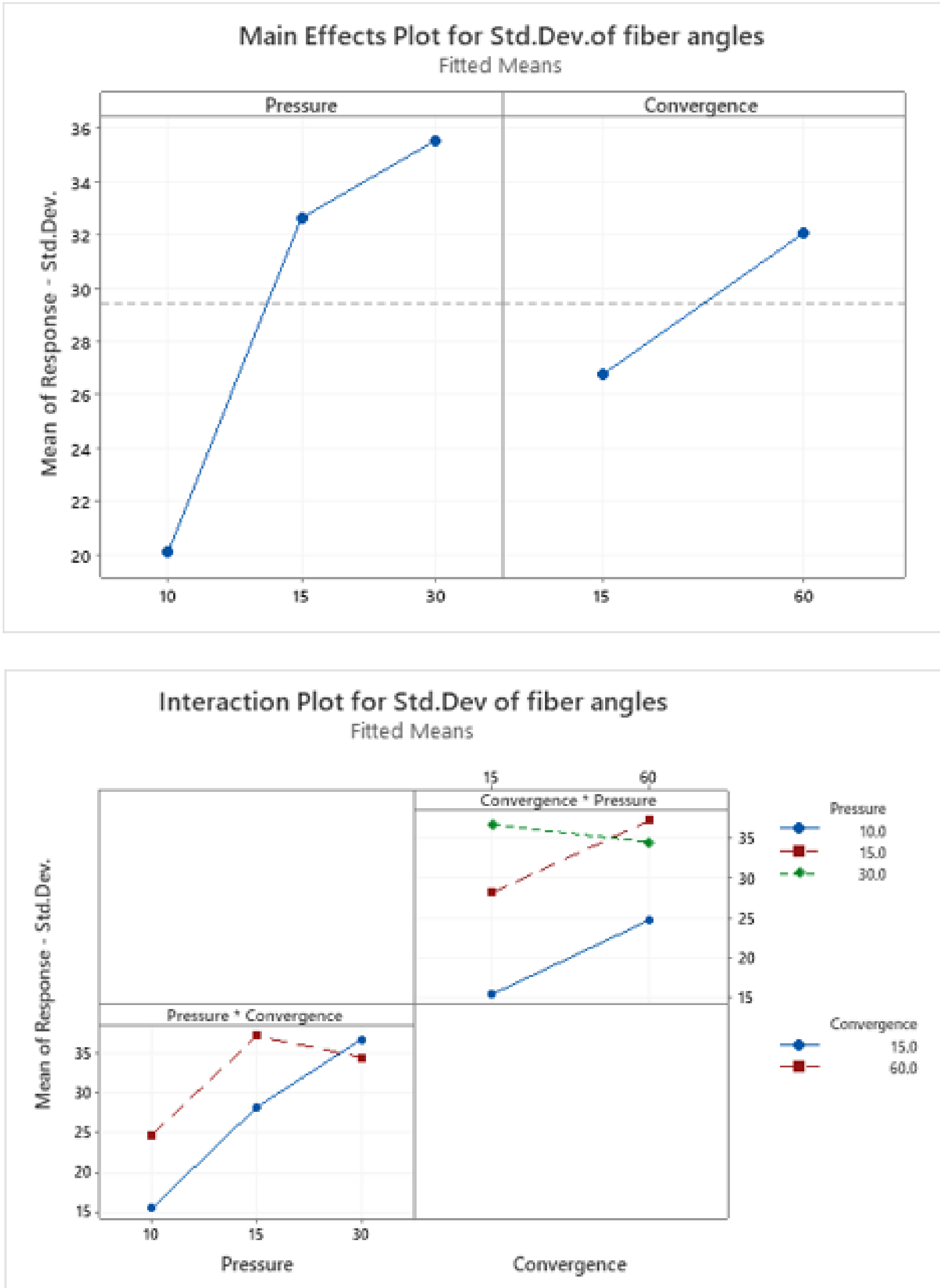

Figure 4.7 Factorial analysis effects plots for standard deviation as response (angles recorded with sign convention)

4.4. Discussion

The initial hypothesis about higher extrusion pressure leading to higher degree of alignment failed after the experimental analysis. This can be explained by reflecting on the assumptions before the experiments were performed. The first and foremost underlying assumption was that the flow is governed by the primary phase alone which is also assumed to be a Newtonian fluid. In fact, a volume fraction of 9% for sugar fibers is significantly high. The sugar fibers present in the bulk also contribute to the nature of flow and hence this is a multi-phase flow system. The traverse velocity calibration curve (Figure 2.4) shows that the traverse velocity does not vary linearly with applied pressure in the same geometrical conditions of the extrusion nozzle. This indicates that the material potentially has a shear thinning or shear thickening property. The shear rate in any cross section of the flow of material cannot be assumed to be smoothly varying because as the flow encounters fibers in its way, the shear rate is distorted because of the fiber-resin interaction.

During extrusion experiments, it was observed that, after extrusion, the resin flows out of the printed sample leaving the sugar fibers at deposition location. Needle diameter used was gage 14 (1.6 mm) but the width of printed sample is almost twice as the needle diameter. This is due to the stress release in the material as it is pushed through smaller cross section area into open atmosphere. Even though the resin itself can be assumed to be incompressible, the mixture of sugar fibers and resin is not completely incompressible in nature and hence the fibers are allowed to move even after extrusion. Thus, further careful material analysis is required to understand this process of fiber-resin extrusion.

4.5. Dissolving sacrificial material

To dissolve the fibers from cured samples, a hot IPA bath of 70°C was used. This is the same technique used by Bellan et.al [10]. Although this method worked very well for the casted samples, it does not work with the extruded samples.

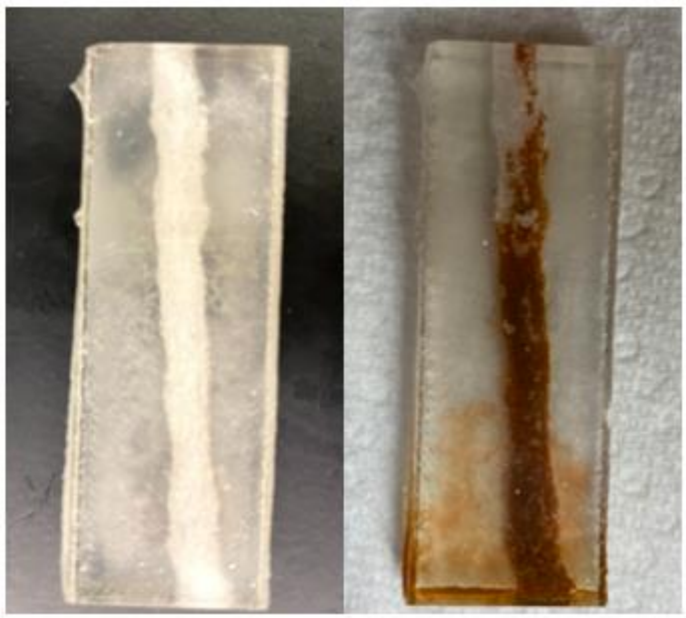

Figure 4.8 (a) Casted sample with sugar fibers (b) Sugar fibers dissolved - dye has penetrated through the channels formed

In case of printed samples, the dissolved sugar stays in place inside the channels and once removed from the bath, it recrystallizes to occupy the channels. In other cases, it was observed that there was not enough area exposed to the solvent which prevented or delayed the process of dissolving fibers. An effective way to dissolve and remove sacrificial material needs to be researched and developed for these shortcomings.

5. CONCLUSION

The idea of mixing sacrificial fibers with curable base to produce micro-channels is feasible with certain practical limitations. Uniform mixing of the fibers without breaking them, obtaining consistency in the mixture in terms of viscosity for extrusion and identifying the right combination of pressure and convergence angle for a defect-free print are the key challenges in this direction. Material compatibility study also needs to be performed to identify a suitable pair of sacrificial and base material. Once printed and cured, dissolving the sacrificial material poses the next challenge. Maximizing area of exposure to the solvent and assuring interconnectivity of extruded fibers will resolve the dissolving issues. Based on the experimental challenges faced during this study, below are key learnings –

- Extrusion of fiber and base mixture is not governed alone by the primary phase in the mixture. It is a multi-phase flow phenomenon that needs to be analyzed by considering the fiber-base interactions during the flow.
- Desired properties for sacrificial fiber material include high fiber strength, high elasticity, chemically inert to base material
- Fiber dispersion in the mixture, interconnected fibers and high exposure area to solvent are the key factors for creating channels after curing
- More research needs to be done for identifying the right material combinations and the printing parameters for each of them; no two material combinations are expected to have the same effect on alignment with printing parameters due to different fiber-base interactions in the flow.

A full-factorial experimental design was evaluated for understanding the effect of extrusion pressure and the angle of convergence of nozzle/extruder on the alignment of fibers in a fiber-resin mixture. This study included experimental work related to finding out optimum sugar fiber proportion for extrusion with Sylgard 184; exploring different techniques for fiber dispersion in the resin; calibration of traverse speed for different extrusion pressures and capturing alignment of each experimental condition.

Following are the major findings of this study:

- Optimal proportion of sugar fiber with Sylgard 184 for syringe extrusion is ~9% by volume (assuming bulk density of sugar = 0.85g/cc)
- Manual mixing is the most effective way for sugar fiber dispersion without causing damage to the fibers. This can be automated by quantifying applied force and controlling that amount in the automated method
- Pressure and convergence angle – both affect the degree of alignment of fibers in extruded mixture. Pressure is more dominant factor compared to the angle of convergence.
- The effect of pressure on alignment is affected by the angle of convergence at higher values – it is a coupled effect.
- Results for alignment are found to be consistent in both the analysis methods – standard deviation as well as mean of the absolute angles
- Dissolving sugar fibers in casted samples is much easier than that in extruded samples due to low exposed area of the sugar fibers to solvent, inconsistent network connectivity of fibers and volume density of fibers in the bulk

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