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ANALYZING THE EFFECT OF ADDITION OF SS 316L NANOPARTICLES ON THE HARDNESS AND IMPACT STRENGTH OF 3D PRINTABLE PMMA RESIN

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ABSTRACT

The mechanical properties of a 3D-printed object are significantly affected by both its material composition and preparation parameters. In this study, a 3D printable nanocomposite was prepared from PMMA and SS 316L nanoparticles. The central composite design (CCD) approach of Response Surface Methodology in Design Expert was used for the design of experimentation. A total of 15 sets of nanocomposite samples were prepared in different compositions and under different preparation conditions. Specimens of hardness and impact strength conforming to ASTM standards were fabricated from a resin 3D printer with employed combinations derived from systematic CCD of the mentioned factors. The maximum hardness of 87 SHD and impact strength of 18.2 J/m were obtained at the filler content of 1% (w/w) and stirring speed of 1100 rpm, while the lowest hardness of 81 SHD and impact strength of 14.2 J/m were obtained at the filler content of 0.5% (w/w) and stirring speed of 800 rpm. The optimal preparation conditions for the PMMA nanocomposite were determined to be 1.05133% (w/w) filler content, 1234 rpm stirring speed, and 30 minutes of sonication time. To validate these findings, the experimental values for Izod impact strength and hardness were determined at the optimized process parameters and found to align close to the predicted values, confirming the reliability of RSM model.

1. INTRODUCTION

Additive manufacturing is a modern fabrication technique used globally in industrial and research applications to fabricate complex 3D objects and geometries. It is a fabrication process that involves a machine driven by a computer, incrementally adding layers of material to create a three-dimensional structure (Jiménez et al., 2019; Ngo et al., 2018). Additive manufacturing uses no tool to fabricate 3D parts; hence it offers excellent features to companies (Prakash et al., 2018). People never thought that experts would use 3D

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printing to make complex parts for airplanes, medicines, fashion, etc. (Haleem & Javaid, 2020; Woźniak et al., 2024). Persevered innovation has pushed 3D printing into new nation-states and proved how long the generation has come because of its invention. 3D printing has many applications in the medical field as these are advanced fabrication techniques based on CAD digital models to form specific 3D objects automatically (Haleem & Javaid, 2019; Javaid & Haleem, 2018). The technocrats can easily access the machines used in medical science with the help of new engineering techniques. Thus, the biomedical field has achieved a spectacular status in recent years. It can fabricate human parts like bones, ligaments, the human heart, skin, drug products, tissues, blood vessels, organs, surgical tools, etc. (Javaid & Haleem, 2020). A few years ago, it was abnormal that a mechanical engineer was working on medical science projects. But today, mechanical engineers are working as well as researching the medical field (Aimar et al., 2019). 3D Printing is often used in orthopedics to make custom implants, tools and devices and It can also be used for planning before surgery and teaching students and patients about the steps. (Andrés-Cano et al., 2021; Meng et al., 2023).

AM uses different methods like Stereolithography (SLA), Selective Laser Sintering (SLS) and Fused Deposition Modelling (FDM) (Woźniak et al., 2022). Each one has its own special benefits and uses (Kafle et al., 2021). This way of making things has made it easier to make complex designs quickly and create specific parts in small amounts. The good thing from AM is the reduction of waste material compared to old ways of making things (Conner et al., 2014). This part about keeping things going green is getting more and more important when talked about the environment. AM can also make difficult shapes and projects that are hard or not possible using normal ways (Nadagouda et al., 2020). Resin 3D printing is a kind of additive manufacturing technology that makes things with a liquid material called photopolymer resins. This uses ultraviolet (UV) light which solidified the resins and make detailed 3d prints (Pazhamannil & Govindan, 2020; Wang & Wang, 2017). This method is liked a lot because it can make things with accuracy and precisely which makes it very useful in many jobs like healthcare and dental sectors. Resin 3D printing is widely used in medicine. It assists in looking after patients, organizing operations and learning about medicine. A key purpose is making body shapes for planning operations before they take place. Doctors can use these clear and detailed models to see complex things, test steps, and improve their surgery before they use the tools. This shortens the time in surgery and improves the patient's outcomes. Moreover, resin 3D printing assists in making unique implants and prosthetics for patients. Made-to-fit implants, designed for someone's body shape, offer a better fit. This increases the way they function and their ease. This change is great in orthopedics because parts can be made to match the unique shapes of a patient's bones (Jandyal et al., 2022; Kholgh Eshkalak et al., 2020; Lin et al., 2019; Tian et al., 2021). In health studies, 3D printing with resin is used to make models for drug tests. This aids scientists in knowing how medicines and treatments work safely. Organ-on-a-chip systems also use this tech. It can improve and make accurate the process of creating drugs (Bloomquist et al., 2018; Lim et al., 2018).

The dental sector is using resin 3D printing for many things, changing old ways of making dental parts (Bogdan & Michorczyk, 2020). An important use of it is for making dental crowns, bridges and other artificial parts for teeth. Resin 3D printing helps make dental models that are very accurate and detailed, allowing dentists to create specially made crowns and bridges that fit each patient's mouth perfectly (Park et al., 2020; Rezaie et al., 2023). Also, this technology is commonly used to make clear braces for dental treatment. Resin 3D printing make exact braces and these braces slowly move the teeth to the right place. This way gives a better feeling and looks nicer than the usual braces (Pillai et al., 2021; Tartaglia et al., 2021). Dental sector use resin 3D printing to make fast replicates of dental tools and models. This lets them check and change things easily before making the final products. The skill to create detailed dental parts means the fixes fit well in a person's mouth and work at their best (Al Wadei et al., 2022; Park et al., 2020). Polymethyl methacrylate (PMMA) is a useful material for dental crowns and bridges (Zafar, 2020). It works very well in 3D printing. Its natural fit with our mouth parts makes it safe to use instead of dental prosthetics. The non-toxic nature of PMMA helps in making pretty dental restorations that look just like real teeth (Hassan SMS, 2019; Mansoor et al., 2022). Its thin texture and easy movement make it good for 3D printing. This helps create complex dental shapes quickly and very accurately. It gives helpful and attractive solutions to patients while they wait in the meantime (S. G. Chen et al., 2019; Dimitrova et al., 2022). To fix worries about being strong, using fillers in PMMA is a good idea. By adding fillers like glass fiber, tiny metal powder or carbon nanotubes, the strength of PMMA can be improved. This makes it harder to bend, more flexible, stiffer and lasts longer, resulting an increases the use of PMMA in dental restoration over time and making PMMA a strong material in today's dentistry (S. Chen et al., 2018; Gad & Abualsaud, 2019; Lyu et al., 2024).

The goal of this present research is to generate a new 3D printable provisional dental restoration-based resin material modified with nano-fillers of medical grade stainless steel and investigate the impact of stirring speed and sonication time at various loading condition of SS 316L nanoparticles. To enhance the understanding and predictive capabilities and optimization Response Surface Methodology (RSM) is used. The results of this investigation are validated using scientific characterizations, testing, and discussions.

2. MATERIAL AND METHODS

2.1 Materials

The dental resin material, PMMA (poly methyl methacrylate), was purchased from 3 Idea Technology LLP (Mumbai, Maharashtra, India). It has a viscosity ranging from 1.15 to 1.20 g/cm2 and comes in a white color. The nano powder used was stainless steel 316L, purchased from Vedayukt India Private Limited (Jamshedpur, Jharkhand, India). This powder has a particle size range of 20-40 nm and a purity level of 99%. The ethanol and Isopropyl alcohol ((CH3)2-CH-OH) with purity 99.9 % was purchased from Science Sales (Sonipat, Haryana, India). All the materials that were obtained, were used without any alteration and surface modification.

2.2 Nano-Composite Preparation

The Design Expert13 software was used for design of experimentation. Three factors were taken based on the literature, and their levels were decided on the basis of pilot experimentation and literature [36, 37]. A total of 15 sets of combination of experiments were obtained from central composite design from three factors and their levels. The fabrication process of the nano composite involved incorporating nanopowder at various concentrations (0.5% (w/w), 1.0% (w/w), and 1.5% (w/w)) under specific working parameters using a hot plate magnetic stirrer and a probe sonicator. The nanopowder was first cleaned in ethanol to ensure purity. For accurate measurement, a weighing scale with a least count of 0.0001g was utilized to weigh the nanopowder samples. Subsequently, the dispersion process commenced by gradually adding the nanopowder into the resin material while subjecting it to hot plate magnetic stirring for a duration of 120 minutes. The homogeneous dispersion and assimilation of the nanopowder inside the resin matrix was aided by the stirring method. Following the stirring stage, the sample was sonicated at a frequency of 20 kHz using a probe sonicator. This ultrasonic treatment also assisted in the dispersion and homogeneity of the nanoparticles, allowing for their uniform distribution throughout the resin substance. The nano composite was synthesised using this process, providing excellent dispersion of the nanopowder inside the resin matrix, which is critical for getting optimal material characteristics and performance.

2.3 Design and Fabrication of Specimen

All specimen geometries and sizes were developed using solid works software in accordance with ASTM standards. The ANYCUBIC Photon Mono X 6K 3D printer was used for the fabrication process, as illustrated in Figure 1. With an exposure duration of 4 seconds, a bottom exposure time of 50 seconds, a layer height of 50 microns, and a light off time of 0.5 seconds, the printing parameters were kept constant throughout the printing process. Once the printing was complete, the printed specimens were carefully removed from the build plate, and proceeded to remove any support material. The specimens were washed in an IPA (Isopropyl Alcohol) solution for 10 minutes. This step helped in removing excessive resins and ensuring the cleanliness of the printed parts. Then the samples were left to cure for 40 minutes in a curing unit.

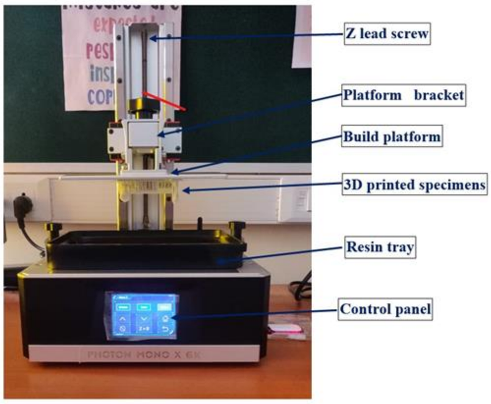

Figure 1. Depicts a resin 3D printer & its components during the fabrication process

2.4 Mechanical Testing

The izod impact test was performed on the specimens fabricated as per ASTM D256 standards, with dimensions of 12.7 mm × 10 mm in cross-section and 63.5 mm in length. The notch of 45 degrees with 2.5 mm depth is located at a distance of 31.8 mm from its one side along the width. The impact strength was measured by placing the specimen in the fixture using the izod charpy impact testing machine (Presto Impact tester, shown in Figure 2).

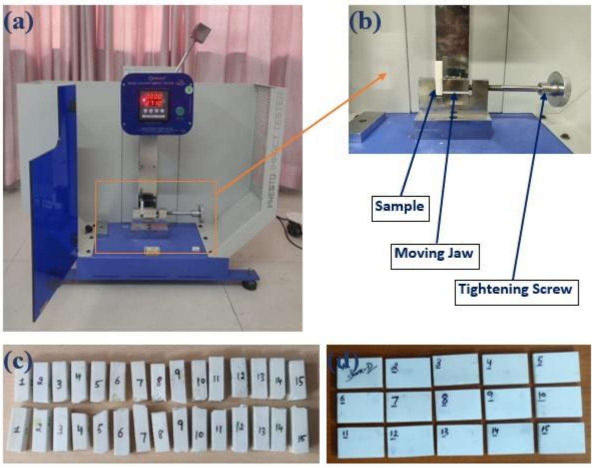

Figure 2. Mechanical testing setup for (a) and (b) izod impact strength, c) izod impact strength tested specimens for d) shore hardness tested specimens

The Shore D hardness for every composite group (n = 6) was determined on cuboidal-shaped specimens (40 × 20 × 6mm) according to ASTM D2240 using a manual analog durometer (MGW Precision Tools, HBD 100-0). The samples were put for 10 seconds under the indenter at multiple separate points along the line for measurement, and mean values were determined. The values of hardness were examined on 0-100 scale, with greater values representing a harder material.

2.5 Statistical analysis

The data was collected from experimental results and statistically analyzed using Design-Expert 13. Box-Wilson or Center composite design was used to tabulate the combination of input parameters at their different levels. Various statistical characteristics, such as lack of fit, coefficient of variation, and estimated and modified numerous correlation coefficients, were carefully evaluated among various polynomial models to determine the most suitable model with a statistical significance of $p \leq 0.05$. To understand the effect of input factors on responses, one factor, interaction graphs, and 3D response graphs were generated using Design Expert13 software.

3. RESULTS AND DISCUSSION

In the experimental phase of this study, the izod impact strength test and shore D hardness test were conducted on composite resins commonly used in dentistry. These composite resins were prepared by dispersing nano-fillers with the resin in varying proportions and employing different stirring speeds and sonication times during the preparation process. In compliance with ASTM prerequisites, the composite resin specimens were made using the ANYCUBIC Photon Mono X 6K resin 3D printer. To evaluate their impact behaviour, the notch thickness, cross sectional area of samples, and angle of rise of hammer were examined that provided insight into the samples' performance during testing. Meanwhile, the data collected during the impact test and the shore hardness test was utilized to calculate the impact energy the hardness of the specimens respectively.

Table 1. Responses of izod impact strength and shore hardness**.**

Std	Run	Factor 1 A: Reinforcement Material (% (w/w))	Factor 2 B: Stirring Speed (rpm)	Factor 3 C: Sonication time (min)	Response 1 Izod Impact Strength (J/m)	Response 1 Shore D Hardness (SHD)
3	1	0.5	1400	30	15.2	82
7	2	1	800	20	17.3	85
15	3	1	1100	20	17.4	85
6	4	1.5	1100	20	16.1	85.5
1	5	1.5	1400	10	15.3	84
4	6	0.5	800	10	14.2	81
13	7	1	1100	20	17.9	85
12	8	1	1100	20	17.3	85.5
5	9	0.5	1100	20	14.8	81.5
14	10	1	1100	20	17.5	86
11	11	1	1100	20	17.4	85.5
2	12	1.5	800	30	15.8	85
9	13	1	1100	10	18.2	85
10	14	1	1100	30	18.1	87
8	15	1	1400	20	17.6	86

An impact tester was used to evaluate the impact energy of the 3D-fabricated test samples. As shown in Table 1, a total of 15 measurements were collected to establish a test configuration for evaluating the impact energy of 3D produced test parts with different composition and processing parameters. The amount of nano-filler, stirring speed, and sonication duration are among the factors. As seen in equation (1), the relationship between the input components and the impact energy is given by a quadratic equation with two-factor interaction.

Izod impact strength = 1.25 + 23.32843A + 0.011036B – 0.221912C – 0.002833AB + 0.0050AC + 0.000075BC – 9.50588A2 – 4.1830 × 10-6 B2 + 0.3235C2 (1)

The investigation found that the impact strengths of the samples varied based on the processing settings. The maximal impact strength of a sample (Run 13) with 1% (w/w) reinforcement, a stirring speed of 1100 rpm, and a sonication period of 10 minutes, for example, was 18 J/m. Sample (Run 6) with 0.5% (w/w) reinforcement, a stirring speed of 800 rpm, and a sonication period of 10 minutes, on the other hand, exhibited a minimum impact strength of 14.2 J/m. In comparison, sample (Run 12), which contains 1.5% (w/w) reinforcement and was made at an 800 rpm stirring speed and 30 minutes of sonication period, had an impact strength of 15.8 J/m. An impact tester was used to measure the impact strength of each test specimen, and the average findings are shown in Table 1.

The shore hardness of samples was subjected to testing using manual analogue durometer. The hardness was calculated at five different points in a row on each specimen. Each specimen exhibited varied values depending on the factors involved, and the absolute computed value was recorded as the response for each test run, as illustrated in Table 1. The connection between the input factors and the shore D hardness is expressed through a quadratic equation incorporating two-factor interaction, as denoted in equation (2).

Shore hardness = 0.74.15033 + 16.92157A + 0.002908B - 0.253922C + 0.003333AB + 0.100000AC + 0.000167BC - 9.29412A2 - 3.59477 × 10-6 B2 + 0.001765C2 (2)

3.1 Validation of Model

Response Surface Methodology (RSM) is a powerful statistical technique used in experimental design, analysis, and optimization. By fitting the data to this mathematical model, RSM helps to gain insights into the interactions among multiple variables and identify the optimum settings that maximize or minimize the response. The key principle behind RSM is the assumption that the response variable can be approximated by a polynomial function of the input variables. The effect of independent variable reinforcement material (A), stirring speed (B), and sonication time (C) is represented in Table 1. The regression equations of the responses are computed from the experimental data in quadratic form with two factor interaction, as mentioned in equations (3) and (4).

Table 2. Regression coefficient values for nano-composite resin

Regression Coefficients	Izod Impact Strength (MPa)	Shore D Hardness (SHD)
Intercept	17.65	85.59
A-Reinforcement material	0.65	2.00
B-Stirring speed	0.15	0.50
C-Sonication time	-0.05	1.00
AB	-0.425	0.50
AB	0.025	0.50
BC	0.225	0.50
A^2	-2.38	-2.32
B^2	-0.3765	-0.3235
C^2	0.3235	0.1765

Based on the ANOVA results, it was found that the experimental data can be effectively represented by a quadratic polynomial model. The coefficient of determination (R2) values for impact strength (R1) and hardness (R2) were determined to be 0.9907 and 0.9685 respectively, as shown in Table 2. Furthermore, the lack of fit was not statistically significant ($p \leq 0.05$) compared to pure error for all variables, suggesting that the model is statistically accurate in representing the data.

3.2 Effect of Process Parameters on Responses

The izod impact strength of composite resin is dependent on the amount of reinforcement material due to its significant effect at linear, quadratic, and interaction levels with sonication time and stirring speed with $p < 0.05$ for all factors. The other factors which also affected the impact strength slightly, were the linear and interactive terms of stirring speed and sonication time. Overall, Fig. 3 (a), (b), and (c) illustrates the relationship between the independent variables (sonication time, stirring speed, and reinforcement material) and the dependent variable (impact strength), allowing us to visually understand how these factors affect the impact strength of the composite resin. The observations indicate that the impact strength shows an increase when the reinforcement material is increased up to 1 percent. However, if the reinforcement is further increased, the impact strength begins to decrease. Additionally, a slight increase in the impact strength is observed at stirring speed nearly 1200 rpm, while sonication times affects the impact strength negatively at initial stage, but gradually increases with increase in sonication time. The results demonstrate a significant and favourable influence of the process parameters on the impact strength.

The hardness of dental restoration is an important factor to consider when choosing a suitable material for a patient. Dental crowns are subjected to forces during chewing and biting, and their durability is crucial for long-term success. The hardness of a dental crown depends on the material used and its topology. In concern to this PMMA composite dental resin, the amount of reinforcement material had a pronounced effect on hardness at linear ($p < 0.001$) and quadratic levels ($p < 0.05$). Other factors which significantly affect the hardness were sonication time, the interaction of reinforcement material with stirring speed, and sonication time. Figure 3 (d), (e), and (f) enable us to visually interpret the effects of the independent variables on the dependent variable, helping us to make conclusions about how sonication time, stirring speed, and reinforcement material affect the specific hardness of the composite resin. The data demonstrates that the hardness declines as the reinforcement material increases to 1.2 percent. However, any additional increase in the reinforcement material slightly decreases it. Additionally, the hardness is minimally influenced by the stirring speed and sonication time. The highest hardness of 87 SHD was observed at an 1100 rpm stirring speed, 30-minute sonication time, and 1 percent reinforcement. These findings demonstrate a significant influence of the process parameters on the hardness.

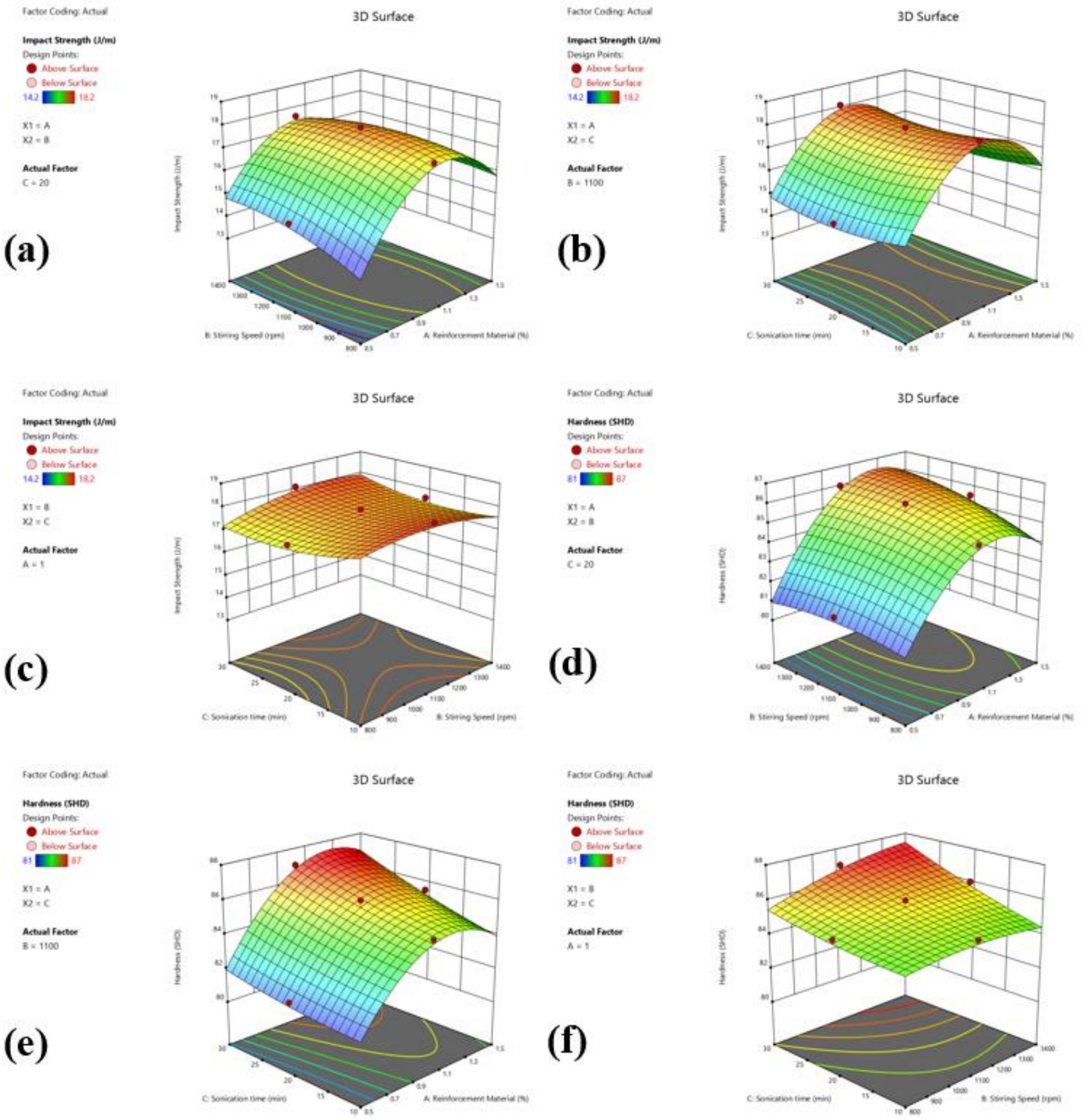

Figure 3. 3D graphic surface optimization of (a) impact strength versus reinforcement material and stirring speed; (b) impact strength versus sonication time and reinforcement material; (c) impact strength sonication time and stirring speed; (d) hardness versus reinforcement material and stirring speed; (e) hardness versus sonication time and reinforcement material; (f) hardness versus sonication time and stirring speed

Further research on the composite material may be conducted by increasing the granularity of the reinforcement increments by 0.1 percent, which would offer a more thorough knowledge of the interaction between reinforcement material and mechanical characteristics. This method would enable a more detailed investigation of the link between variables and responses, perhaps uncovering trends or patterns that would not be apparent with bigger increments. Researchers may acquire deeper insights into the ideal range of reinforcing percentages for attaining desired mechanical characteristics in composite resin by discovering these finer characteristics. Researchers may deepen their knowledge of the relationship and make more informed judgements in designing and creating more effective and durable composite materials by broadening the range of reinforcing increments.

3.3 Optimization of Process Parameters and Validation

In this study, the process factors were optimized by implementing RSM (Response Surface Methodology). The primary objective was to enhance the hardness and izod impact strength. To achieve this, a multi-objective optimization approach was employed to determine the optimal combination of process parameters that would yield the best outcomes. The specific requirements for the multi-objective optimization, including the responses, shore hardness and izod impact strength, are provided in Table 3.

Table 3. Requirement for Multiple-Objective Optimization

Name	Goal	Lower Limit	Upper Limit	Lower Weight	Upper Weight	Importance
A: Reinforcement material	within limits	0.5	1.5	1	1	***
B: Stirring speed	within limits	800	1400	1	1	***
C: Sonication time	within limits	10	30	1	1	***
Impact strength	maximize	14.2	18.2	1	1	***
Hardness	maximize	81	87	1	1	***

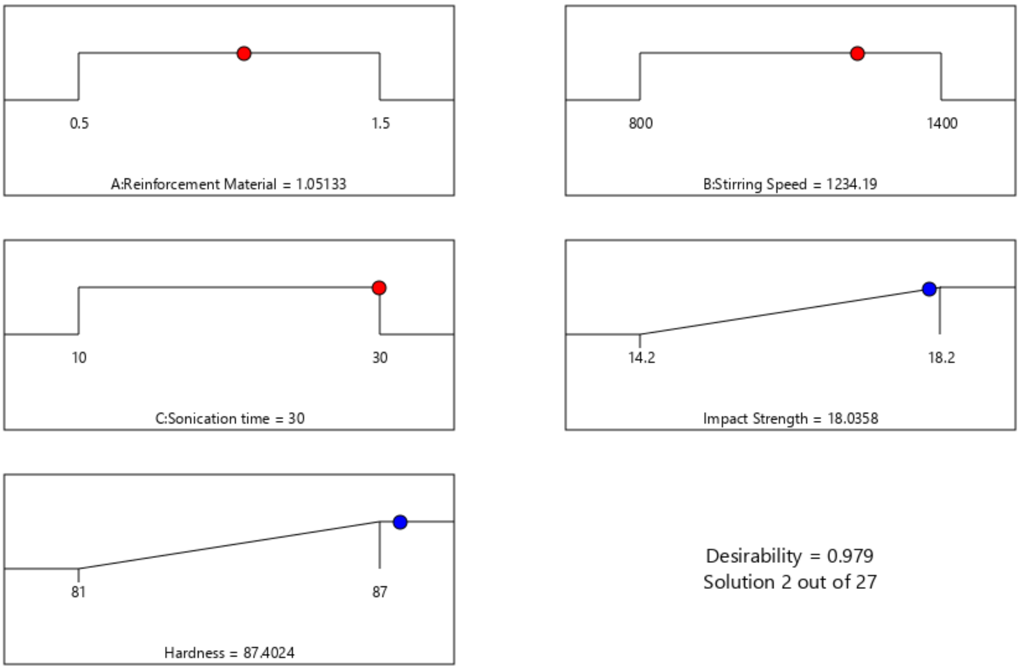

Figure 4. Ramp function graphs for Multiple-objective optimization represent input independent factors' values and responses at optimized state with maximum desirability

The results are displayed in the ramp graph in Fig. 4 with maximum desirability. The optimum process parameters – reinforcement material, stirring speed, and the sonication time - found to be 1.05133% (w/w), 1234.19 rpm, and 30 min, respectively, were needed to achieve the maximum izod impact strength and hardness simultaneously. The predicted impact strength and hardness values are 18.0358 J/m and 87.4024 SHD, respectively, at the optimal set of parameters. The model was validated using the best possible process parameter combination revealed by the RSM-generated models at maximum desirability. To verify the accuracy of the projected models, a minimum of three specimens have been made using the best process parameter combination chosen for each magnitude. It is confirmed that the generated RSM model is adequate when the observed average minimum 0.12026% variation in izod impact strength and 0.05321% variation in shore hardness was near the expected values of 0.15321% and 0.05932%, respectively.

4. CONCLUSION

The study introduced the preparation condition of 3D printable resin reinforced with SS 316L nano-fillers using magnetic stirring and probe sonicator in order to analyze izod impact strength and hardness. The design of experiments was obtained using the CCD approach, and RSM was used to maximize the izod impact strength and hardness. A quadratic model was obtained for describing and forecasting the responses of impact strength and hardness, with the change of independent factors (nano-filler content, stirring speed, and sonication time). In comparison to unmodified resin material, the incorporation of SS 316L nanoparticles increased the resin's impact strength and hardness. Optimized preparation conditions for nanocomposite resin were 1.05133% (w/w) of reinforcement material SS 316L, a stirring speed of 1234.19 rpm, and a sonication time of 30 minutes, resulting in the predicted impact strength and hardness values 18.0358 J/m and 87.4024 SHD, respectively. These values were found to closely align with the predicted values which confirms the reliability and accuracy of the developed RSM model, making it a valuable tool for optimizing the preparation conditions of nanocomposite resin and identifying the relation among the independent factors and responses. The newly created 3D printable nanocomposites could be more effective and have a higher potential as long-term temporary dental restorative materials.

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