COMPARISON BETWEEN INJECTION MOULDED FLEXIBLE DENTURE BASE MATERIAL AND NOVEL 3D PRINTABLE NYLON RESIN IN RELATION TO SURFACE ROUGHNESS AND FLEXURAL STRENGTH – AN IN VITRO STUDY

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Abstract

The aim of the study was to compare and evaluate the flexural strength using universal testing machine and surface roughness using surface profilometer of Injection Moulded flexible denture base material and 3D printed Nylon resin. A total number of 60 samples was divided into 2 groups, 30 for each material and further subdivided into 15 each for testing of flexural strength and surface roughness. The average flexural strength of the 3D printed group was higher than that of the injection moulded group which were 0.6720KN and 0.6027KN respectively while the average surface roughness of injection moulded group was higher than that of the 3D printed group which were 2.37793µm and 2.0893µm respectively with a statistical significance value of 0.001. It was concluded that 3D printed nylon resin samples have comparable flexural strength to the injection moulded samples but a lower surface roughness when compared to the injection moulded samples.

Keywords: Removable partial denture, Flexible dentures, CAD-CAM, 3D printing, Nylon

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1. Introduction

Modern dentistry presents with a wide variety of options for the restoration of partial edentulous mouths, including removable partial dentures (RPDs), fixed partial dentures and dental implants. The metallic appearances of the clasp in conventional cast partial dentures may be disadvantageous, while treating the patients who are very much concerned about the aesthetics.[1] Polymerization shrinkage encountered in conventionally cured PMMA made way for the development of a special injection-moulding technique. Advantages of injection moulding include elimination of flash of resin between the halves of the flasks, and minimal polymerisation shrinkage. However, certain drawbacks are associated with the injection moulding technique include which the fracture of the material and presence of cracks.[2] One of the main disadvantage of PMMA includes the low flexural strength and surface hardness.[3] Nylon based flexible resins were developed for the construction of provisional prostheses such as immediate RPDs, and are indicated for the construction of RPDs, mainly for anterior retention with esthetic requirements, due to the advantages of translucency and a natural appearance without laboratorial characterization. Additionally, the flexibility of these materials prevents the fracture of prosthesis and permits them to be lighter and more comfortable. Flexible RPDs require no tooth preparation as do conventional RPDs, and they reduce the chair time required to construct the prosthesis.[4] Intraoral scanners and computer-aided design and computer-aided manufacturing (CAD-CAM) technology have been widely used for fabricating fixed partial dentures and complete dentures. However, less has been published concerning use of digital and CAD-CAM technology for removable partial dentures. Removable partial dentures are difficult to fabricate by using CAD-CAM because they are made from several parts, a metal framework, a denture base, and artificial teeth.[5]

Studies describing 3D printable Polymethyl Methacrylate (PMMA) for removable partial and complete dentures have shown improved properties such as adaptability, durability and lesser volumetric shrinkage.[6] The method of 3D printing however has not been explored for fabrication of flexible denture bases. Stereolithography (SLA) is a commonly used method of 3D printing and was one of the first methods of additive manufacturing to be developed.[7] 3D printable Nylon-Green Tough Resin consists of Nylon 6 as resin additive. Nylon 6 usually has different ordered crystalline phases in its matrix resulting in non-crystalline/amorphous regions and a dominant factor of the mechanical property behaviour. Other additives include colour pigments and nanomaterials for improved resin performance.[7] These resins are stretchable, withstanding high pressures up to 19 MPa, and do not break easily and are specifically made for prototyping and creating functional resin pieces.[8]

Various studies have proved that PMMA has smoother surface compared to polyamide nylon resins for flexible denture base.[9] This may be due to the difficulties associated with finishing and polishing of nylon denture base materials. 3d printing has shown to render smoother surfaces compared conventional injection moulding techniques for denture base materials. Flexural strength is one of the important mechanical properties which is usually tested for flexible denture base materials.[10] Hence; under the light of above-mentioned data, this study was planned to compare and evaluate the flexural strength and surface roughness of Injection Moulded Valplast (flexible denture base material) and 3D printed Phrozen Nylon resin.

The objectives of the study included

- To evaluate and compare the surface roughness between flexible denture base material (Valplast) and Nylon tough 3d Printed resin (Phrozen) in Microns using Mitech surface roughness tester MR200 , according to ISO 20795-2:2013

- To evaluate and compare the flexural strength between flexible denture base material (Valplast) and Nylon tough 3d Printed resin (Phrozen) in MPa using 3 point bending test in Universal Testing Machine according to ISO 20795-2:2013

The null hypothesis stated was, 'there is no difference between flexural strength and surface roughness between the injection moulded samples and 3D printed nylon resin samples."

2. Materials and Methods

A total number of 60 samples was divided into 2 groups. Group 1 consisted of 3D Printable Nylon Tough Resin samples (Figure 1) and group 2 consisted of . Injection Moulded Flexible Nylon Resin samples. The two groups were further subdivided as Group 1A, 1B, 2A and 2B. Group 1A consisted of 15 samples of 3D printable nylon resin for testing surface roughness and group 1B consisted of 15 samples for testing flexural strength. 2A consisted of 15 samples of Injection Moulded flexible resin for testing surface roughness, and 2B consisted of 15 samples for testing flexural strength.

Preparation of 3D printed Nylon Samples for surface roughness and flexural strength

Cuboidal samples of dimension 65mm x 40mm x 5 mm (lxbxh) according to ISO 20795-2:2013 were designed in a CAD software Autodesk 123D design. 30 such samples were 3D printed with the Phrozen Nylon Tough resin (Taiwan) using X3D V1 3D printer (Align It, India) (Figure 2). The sample was post cleaned with 99.9% isopropyl alcohol in an ultrasonic cleaner (Unident, India) for 5 to 7 minutes and post cured in a UV curing chamber (X3D V1, Align It, India) at 405nm for 5 to 7 minutes.

Samples were finished with

1. Abrasive paper (Cumi, India) grit size 80 to 1200 with light manual pressure.
2. A slurry of medium grit pumice mixed in a 1:1 ratio of water will be used.
3. A cloth wheel of 12.5 µm for 60 s at 3,000 RPM on the polishing lathe. This will be repeated with fine grit pumice (Neelkanth, India)
4. A second cloth wheel (Polirapid, Germany), high shine buff will be used with polishing brown Tripoli for 60s

Preparation of Injection Moulded Nylon Samples for surface roughness and flexural strength:

A master die of dimension 65mm x 40mm x 5 mm (lxbxh), ISO 20795-2:2013, was designed in a CAD software Autodesk 123D design and milled in Grade 304 stainless steel BMC 650L milling machine (India). Paraffin oil (Zen Vista Meditech, India) was applied on the surface of the die. Hindustan Modelling wax No. 2 (India), was heated using ALE Analogue wax pot 3 (Unident, India) and poured into the master die. The wax was allowed to cool at room temperature for 30 minutes and removed carefully with finger pressure. 30 such wax patterns were fabricated and invested immediately on a flexible denture flask with Type II Gypsum (DPI Maharashtra India). Sprue formers were attached to make the channels for flowing of fluid resin into mould. The flexible denture flask was allowed to set at room temperature for 30 minutes followed by dewaxing at 100 degrees C. Dewaxing was done by placing flasks in boiling water for 3 to 5 minutes to soften the wax in dental dewaxing bath (Unident, India). The flask was opened flushed with clean boiling water to remove all the residue of wax. The flask margin was checked to and ensure that both flask halves fit together with intimate metal contact. A thin coat of silicon spray (Aditya Silicone, India) separating agent was applied to model and the model was allowed to dry completely.

Specimens were processed using a microinjection system (Sabilex 2AD Plus) according to instructions provided by the manufacturer. In this system, the high pressure of the injection process accurately regulates the inflowing amount of material into the closed dental flask. The material was plasticized for 15 to 20 minutes at 287 to 293°C. An aluminium injection cartridge of Valplast (USA) was used to carry the mixture for injection. The injection process was carried out in the injection unit for 1 min at a pressure of 2 bars. The pressure was maintained for 3 to 5 minutes. The pressure was then relieved, and the flask was allowed to bench cool for at least 15 to 20 minutes before opening. The sprue formers were cut with special type of knife or disk and finishing with:

1. Abrasive paper 80 grit to 1200 grit with light manual pressure.
2. A slurry of medium grit pumice mixed in a 1:1 ratio of water will be used.
3. A cloth wheel of 12.5 µm for 60 s at 3,000 RPM on the polishing lathe. This will be repeated with fine grit pumice.
4. A second cloth wheel, high shine buff will be used with polishing brown Tripoli for 60s

All the samples were stored in normal saline for 48 hours before testing.

Evaluation of Surface Roughness:

The two groups, Group 1A and 2A were tested for surface roughness using the Mitech Surface Roughness Tester MR 200 (India), with resolution 0.01µm ± 0.002µm (Figure 3).

Evaluation of Flexural Strength:

The two groups 1B and 2B were tested for flexural strength in 3-point bending test using Universal Testing Machine Model UTN 40 SR NO. 11/98-2450 with a process speed of not more than 5mm per minute. (Fuel Instruments and Engineers Pvt Ltd, Maharashtra, India) (Figure 4).

All the values obtained were statistically evaluated and compared using Unpaired T test, Independent Sample Test using the SPSS software.

Figure 1. 3D printable nylon resin

Figure 2. 3D Printing of Samples for Evalauting Surface Roughness and Flexural Strength

Figure 3. Evaluation of Surface Roughness

Figure 4. Evalaution of Flexural Strength

3. Results

Table 1. Standard Deviation and Standard Error for Flexural Load

	Group	N	Mean	Std. Deviation	Std. Error Mean

Flexural Load in KN	Phrozen 3d printed group 1	15	.6720	.05213	.01346
	Injection Moulded Group 2	15	.6027	.06431	.01660

Table 2. Independent Samples Test for Flexural Strength

		Levene's Test for Equality of Variances		t-test for Equality of Means						
									95% Confidence Interval of the Difference	
		F	**Sig.**	**t**	**df**	**Sig. (2-tailed)**	**Mean Difference**	**Std. Error Difference**	**Lower**	**Upper**
Flexural Load in KN	Equal variances assumed	1.026	.320	3.244	28	.003	.06933	.02137	.02555	.11311
	Equal variances not assumed			3.244	26.850	.003	.06933	.02137	.02547	.11320

Table 3. Standard Deviation and Mean Standard Error for Surface Roughness

	Group	**N**	**Mean**	**Std. Deviation**	**Std. Error Mean**
Surface Roughness in Ra µm	Phrozen 3d printed group 1	15	2.02893	.077847	.020100
	Injection Moulded Group 2	15	2.37793	.218152	.056327

Table 4. Independent Samples Test for Surface Roughness

	Levene's Test for Equality of Variances	**t-test for Equality of Means**

		F	Sig.	t	df	Sig. (2-tailed)	Mean Difference	Std. Error Difference	95% Confidence Interval of the Difference	
									Lower	Upper
Surface Roughness in Ra µm	Equal variances assumed	13.782	.001	-5.836	28	.000	-.349000	.059806	-.471506	-.226494
	Equal variances not assumed			-5.836	17.5	.000	-.349000	.059806	-.474900	-.223100

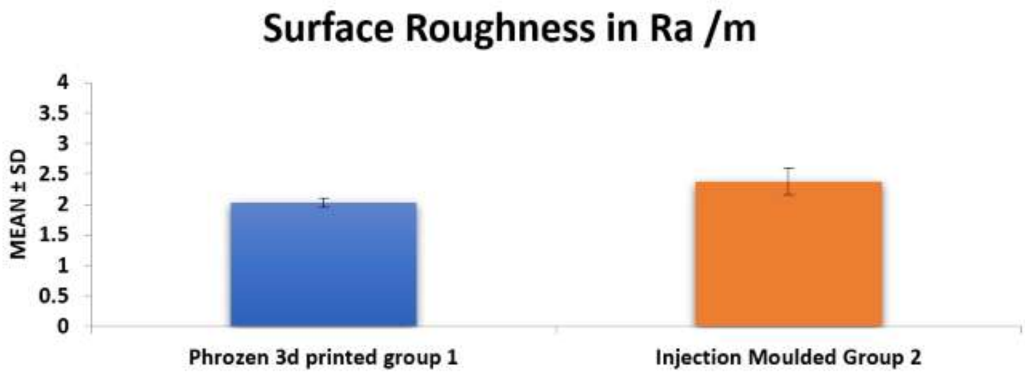

Figure 5. Graphical Representation of Surface Roughness

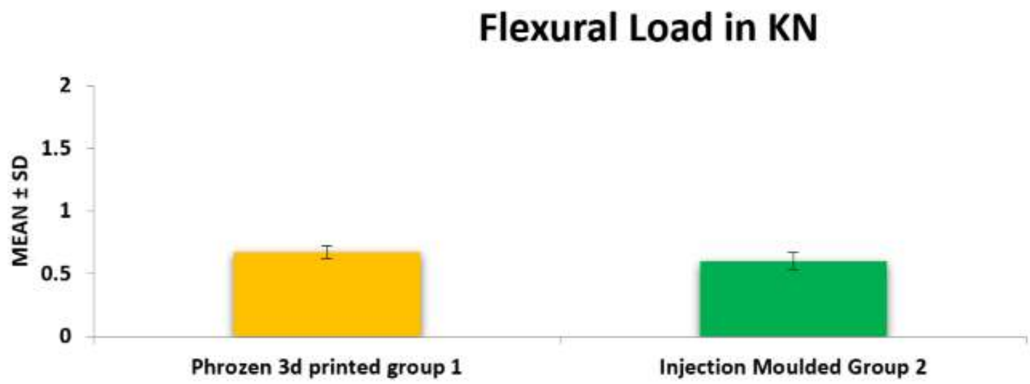

Figure 5. Graphical Representation of Flexural Load

Table 1 represents the mean values of flexural load in KN for conventional flexible denture base material and nylon 3D printed resin. The mean values of the flexural strength of group 1 were higher than the mean values of the injection moulded group 2 which were 0.6720KN and 0.6027KN respectively. This indicates that the flexural strength of 3D printed samples were higher than the injection moulded samples. The standard deviation for 3D printed samples was 0.05213 and the injection moulded samples was 0.6027.

The values were analyzed using independent sample t test and the values are listed in table 2. The F value for the flexural load was 1.026. The statistical significance value was found to be 0.320.Table 3 represents the mean values of surface roughness in µm for conventional flexible denture base material and nylon 3D printed resin. The mean values of surface roughness of injection moulded group 2 were higher than the mean values of the 3D printed group 1 which were 2.37793µm and 2.0893µm respectively. This indicates that the surface roughness of injection moulded samples was higher than the injection moulded samples. The standard deviation of 3D printed samples was 0.77847 and for injection moulded samples was 0.218152.

Figure 5 represents the mean and standard deviation of surface roughness plotted against the two groups of materials. Figure 6 represents the mean and standard deviation of flexural strength plotted against the two groups of materials.The values were analysed using independent t test and the values are listed in table 4. The F value for surface roughness was 13.782. The statistical significance value was found to be 0.001.

4. Discussion

Nylon was introduced as a denture base material in 1955. In the studies by Watt DM, polyamide partial dentures made of nylon 66 as a denture base material were reviewed and it was found that there were some mechanical advantages of the material over conventional polymethylmethacrylate[11]. However it was also found that the material tend to stain and developed a sogginess and roughness of the surface after a few weeks.

Munns D described the use of 100 nylon dentures over a 5-year period and concluded that nylon as a denture base material represented problems such as colour instability and surface roughness[12]. Stafford GD, et al, compared some of the properties of a nylon 12 denture base material with some conventional denture base materials and commercial nylon 12. It was found that the strength of Nylon 12 was considerably greater than other polymers and could be indicated for cases where dentures showed unexplained fractures, or to patients who exhibited allergies to conventional resins[13].Considering the emerging technology of digital dentistry, computer aided design and computer aided manufacturing has been widely used for construction of complete and removable partial dentures. The advantages of stereolithography for 3D printing include better adaptability, durability and lesser volumetric shrinkage. Studies have proven the superiority of 3D printed resins to conventional heat polymerized resins in relation to surface roughness.[14] However flexural strength is found to be dependent on the orientation of the model in relation to the build plate. This study aimed to compare and evaluate the surface roughness and flexural strength of conventional flexible denture base material and nylon 3D printed resin and to evaluate the biocompatibility of the nylon 3D printed resin. The null hypothesis was there is no difference between flexural strength and surface roughness between the two groups. From the results of the study, it can be understood that flexural strength of the 3D printed nylon resin was higher than the injection moulded samples. The surface roughness was found to be higher for the injection moulded group when compared to the 3D printed group. The mean value for flexural strength for 3D printed and injection moulded groups were found to be 0.672KN and 0.602KN respectively. The mean values for surface roughness of the 3D printed group and injection moulded group were2.0893µm and 2.37793µm respectively. These results were similar to the findings by Singh K et al who observed high flexibility of nylon based denture base resins [15]. There was no significant difference between the mean values of the two groups for flexural strength, however statistically significant difference was found for surface roughness. Thus, the null hypothesis was partially rejected. The mechanical properties of 3D-printed resin are affected by build parameters, build orientation, and post-curing process, software, number and thickness of layers, and shrinkage between the layers. The printing parameters are usually set by the manufacturer and cannot be modified except for layer thickness and printing orientation. The acceptable layer thickness is 100 µm and ranges between 25 and 200 µm. However, the strength of the 3D-printed object is increased when the layer thickness decreases due to improved curing of the resin and reduced dimensional changes. Moreover, the geometric details are captured more precisely in thinner layers but the drawbacks include longer processing time, higher cost, and the possibility of print failure. The orientation of the build could also influence the mechanical properties and accuracy of the printed object and the surface finish. It has been reported that vertically positioned object will have higher compressive strength and greater accuracy compared to horizontally position ones[16]. In the present study, the specimens were printed at 90 degrees orientation and 50 micron-layer thickness.There was no significant difference between the flexural strength of the two groups. This result may be due to the material composition, where 3D-printing involves the use of monomer based on esters and has relatively low doublebond conversion compared with conventional resins. Another explanation could be the layering build in a direction perpendicular to the load direction which results in improved mechanical properties due to adhesion between successive layers and the strength within the layer itself. The results are similar to the findings by Shim et al who proposed that samples printed at 45 and 90 degrees showed higher flexural strength than those at 0 degree, this may be due to the fact that specimens built in a direction parallel to the load direction exhibited poor mechanical properties because adhesion between successive layers is weaker than strength within the same layer.[17]

The results of the study are also similar to those by Lee et al who compared the physical property of impact strength of 3D printed Resin to conventional heat cured and cold cured PMMA.[16] The study concluded that 3D printed resin had a greater impact strength than the conventional heat cured resin. However, contradicting results were found in the study by Chhabra et al, who comapared the flexural strength of heat cured PMMA vs 3D printed PMMA.[18] It was found that heat cured PMMA had a greater flexural strength when compared to the 3D printed PMMA. The conventional injectional moulding technique for fabrication of flexible dentures tends to create voids or irregularities in the surface. These may render the denture more prone to microbial adhesion and plaque accumulation. In the studies by Abuzar et al, who evaluated the surface roughness of a polyamide denture base material in comparison with PMMA, it was found that polyamide specimens produced a rougher surface than PMMA, both before and after the polishing process.[19] The unpolished polyamide surface might have been affected by some degrees of disintegration of the mould surface which was heated to a higher temperature compared to PMMA, and also the pressure during injection moulding.

Tripathi et al evaluated and compared the surface roughness in heat cure denture base resins and injection moulded resin system as affected by commercially available denture base cleansers. It was found that The surface roughness increased

significantly in injection-molded polyamide denture-based resin samples when immersed in the denture cleanser.[20]
In the study conducted by Eghtedari et al, while comparing the surface roughness of 2 polyamide materials with conventional heat cured PMMA, it was found that the injection moulded polyamide materials had a higher surface roughness when compared to the conventional heat cured PMMA.[21]
Mekkawy et al compared the surface roughness of different flexible thermoplastic materials before and after polishing. It was found that the surface roughness greatly decreased after polishing.[22]
Priya MSPH et al evaluated the flexural strength of PMMA reinforced with ultrahigh molecular weight polyurethane fibres and teak wood fibres. The results obtained showed similar values to that of the present study, indicating that the flexural strength of the nylon reinforced resin was similar to that of PMMA reinforced with Teak wood fibres.[23]
The present study showed reduced surface roughness for 3D printed resin than injection moulded resin. This may be due to the fact that the printing orientation affects the surface roughness. Printing orientation determines the build direction of layers and the layer-by-layer configuration forming the geometry of the surface. As the surface geometry influences surface characteristics, printing orientation for denture bases would critically affect the surface characteristics of 3D printed dentures. The samples were printed at 90 degree orientation and showed lesser surface roughness. These findings are similar to the study by Shim et al, where it was found that samples printed at 90 degree orientation had least surface roughness and Candida albicans adhesion when compared to samples printed at 0 degrees and 45 degrees.[16] This may be due to minimising the height of step edges and reducing the layer thickness.
The study is valuable in evaluating the flexural strength and surface roughness for the novel 3D printed flexible resin, and proves that 3D printing shows lesser surface roughness when compared to the injection moulded technique. The limitations of the study include the necessity for printing of samples in 90 degree orientation which may not be always possible for clinical situations. The in vitro study did not stimulate the oral conditions such as presence of saliva or oral temperatures. These may affect the mechanical properties of the denture base resins. In order to verify the clinical relevance of this study, follow-up studies with 3D-printed flexible dentures produced in 90 degree printing orientation with stimulation of oral environments are necessary.

5. Conclusion

Based on the findings of this invitro study, the following conclusions were drawn,
1) 3D printed nylon resin samples had comparable flexural strength to the injection moulded samples.
2) The surface roughness of injection moulded samples was higher than 3D printed nylon resin samples. ($P<0.001$)

6. References

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