Journal of
Babol University of Medical Sciences
JBUMS
e-ISSN: 2251-7170 p-ISSN: 1561-4107

Reinforcing Pourable Cold-Cured Resin (in-Vitro) with Micro-Glass Beads

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Article Type
Research Paper

Received:
Aug 23rd 2023
Revised:
Oct 18th 2023
Accepted:
Mar 2nd 2024

ABSTRACT

Background and Objective: Weropress is a pourable auto-polymerising acrylic resin which has recently been used in making dentures. It is characterised by high colour stability and quick setting. However, it is not as strong as heat-polymerised acrylic resin. The objective of this in-vitro study is to find out the effect of adding micro-glass beads on flexural strength, hardness and surface roughness of "Weropress".

Methods: In this study, 24 65×10×3 mm specimens were produced from Weropress following the manufacturer's instructions (casting process). The specimens were divided into 4 groups (n=6): group 1 was "control" which had no additives, group 2; 3; and 4 contained micro-glass particles at ratios 5%, 10%, and 15%. After conventional finishing and polishing procedure, all specimens were immersed in distilled water at room temperature for 2 days. Then, the surface roughness (3 times, 4mm), hardness (four times at 50 g load for 30 sec dwell time), and 3-point bending test (5mm/min speed, 50 mm heads apart) were applied on each specimen and then evaluated.

Findings: Flexural strength reduced for 97 MPa when no filler was added. When 15% filler was added, it reached 75.5 MPa, which had a significant decrease (p=0.03). Other groups showed no significant difference (79.5 MPa and 81.5 MPa). Vickers hardness was fluctuating around (16 MPa) of control following the addition of fillers; however, all testing groups were not significantly different from control. Surface roughness has increased in all groups from 0.53 µm up to 0.77 µm but with no significant difference.

Conclusion: According to results, the addition of micro-glass beads on Weropress powder at 5%, 10%, and 15% has no strengthening effect on Weropress with no important effect on hardness and surface roughness.

Keywords: *Flexural Strengths, Hardness, Cold-Cured Acrylic, Denture Base Resin, Glass.*

Cite this article: Hadi Alabdulla I. Reinforcing Pourable Cold-Cured Resin (in-Vitro) with Micro-Glass Beads. *Journal of Babol University of Medical Sciences*. 2025; 27: e18.

[DOI: 10.22088/jbums.27.1.18]

Introduction

In the recent decade, an auto-polymerising pourable denture material was introduced into the market, called Weropress polymer (Merz Dental GmbH, Germany). This material is characterized by excellent aesthetic properties and a high color stability under different conditions, aging and disinfection, according to previous studies (1-4). This material can be used for constructing partial and complete dentures according to the manufacturer (4). Besides, it can be polymerized in a few minutes as an auto-polymerising polymethylmethacrylate (PMMA) versus several hours for conventional heat-polymerized PMMA material. However, following the polymerization, this material is less strong than heat-polymerized PMMA conventionally used to make dentures and light-polymerized UDMA denture base materials and not ideal for constructing long lasting dentures as confirmed previously (1, 5, 6). This necessitates reinforcing Weropress material to enable taking advantage of its benefits including excellent aesthetics, short polymerization time, and easy manipulation due to the availability of variable methods for handling (Appendix 1).

The reinforcement of acrylic denture base material is a well-known method to increase the mechanical properties of dentures (7, 8). There are so many different reinforcing methods but mainly by incorporating fillers of different shapes, sizes, orientations and forms (9) such as glass fibers (10-12), micron or nano-sized filler of metal oxide, glass, cellulose, or polymer (13) such as ZrO and TiO (14, 15).

Micro-glass particles are inorganic fillers. Few studies have been conducted on testing reinforcement of polymers with glass particles. However, micro-glass particles consist of some components such as sodium oxide, potassium oxide, magnesium oxide, calcium oxide, aluminum oxide and silicon dioxide. Silicon dioxide or silica SiO2 forms the main constituent. The addition of silica particles or silica-based fillers to polymers increases the mechanical properties, including flexural strength and hardness (7, 16-18). They also did not influence surface roughness at low ratios while at high ratios, surface roughness was increased due to agglomeration and crack propagation (8).

There is no research in the literature about the reinforcement of Weropress. The aim of this study is to find out the reinforcing effect of adding different ratios of micro-glass particles on the flexural strength, hardness and surface roughness of Weropress polymer.

Null Hypothesis: (1) Flexural strength and Vickers hardness should increase by the addition of micro-glass beads on Weropress (2). Surface roughness should not change substantially by the addition of micro-glass beads on Weropress.

Methods

This in-vitro study was approved by the Ethics Committee of the College of Medicine/ University of Kerbala with code no. 24-2.

In this in-vitro study, the stone (Dental stone type III, Asian chemicals, India) was mixed and poured into the flask using wax pattern made previously (PremEco Line, MerzDental, Germany) at dimensions (67×10×3.5 mm) (figure 1) according to ISO 20795-1:2013 (19). After setting, the flask was placed in hot water for 5 min to dissolve the wax and then, it was opened. Any remnants of wax were cleaned with hot water to produce the stone mold (figure 2). The stone mold was covered with separating medium (Vaseline) (petroleum jelly, Unilever, India) and used to produce 24 bars from Weropress polymer (MerzDental GmbH, Germany) following the manufacturer's instructions using casting method (Appendix 1). No specimen was disposed of. Specimens were divided into 4 experimental groups (n=6): group 1 is "control" which has no additives on the powder, group 2; 3; and 4 are containing micro-glass beads (Hill-Rom,

[DOI: 10.22088/jbums.27.1.18]

Clinibeads, France) at ratios 5% (1g beads/19g powder), 10% (2g beads/18g powder), and 15% (3g beads/17g powder) in weight, respectively. Electronic balance (Mettler AE 240, Switzerland) with an accuracy of 0.001g was used for weighing the powder and beads.

All specimens were finished using the simple conventional method with a tungsten carbide bur (Dentorium, USA) and low speed handpiece (NSK Kakanishi Inc., Japan). The specimens were then polished using sand paper grit size P320 and P400-grit (3M, USA). After that, the specimens were polished with a cloth wheel and slurry of wet pumice (fine grain size) (Steribim, Metrodent, UK). The thickness and width of each specimen were measured 3 times using a digital Vernier calliper (Mitutoyo, Japan) and the mean value was calculated. All specimens were then numbered and maintained in distilled water at room temperature.

Bar specimens were removed from water following 2 days and dried with a clean tissue. The surface roughness was tested using surface roughness tester (TR200, USA) for the terminal 4 mm of each bar. The test was repeated three times with mean calculation for each specimen in micrometer (µm). Then, hardness tester was used to test the hardness (at the other terminal end of the bar) using Vickers microhardness tester (Laryee, China) at 50 g load for 30 sec dwell time four times for each specimen with mean calculation in megapascal (MPa). Lastly, flexural strength test (3-point bending test) was applied on specimens using Instron machine (Laryee, China) at a speed of 5mm/min, stop head at 20 mm extension and supporting heads were 50 mm apart. The following equation was used to calculate the flexural strength in MPa following ISO 20795-1:2013 (19).

$S=3FL/2bh^2$

F: maximum load (N), L: support span (mm), b: width (mm), h: thickness (mm)

Machines for testing can be seen in the figure 3, 4 and 5 and the specimens in figure 6. Statistical analysis was done using Sigma Plot statistical software version 15.0 at a confidence level of 95% and significance level of 0.05%. Pilot study was done beforehand on 3 specimens from each group to calculate the sample size at 0.95 power. Kruskal-Wallis and one-way ANOVA with Shapiro-Wilk tests were used for analysis.

Figure 1. Wax pattern (bars)

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Figure 2. Stone mold

Figure 3. Vickers hardness tester (Laryee, China)

Figure 4. Instron (Laryee, China)

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Figure 5. Profilometer (TR200, USA)

Figure 6. The specimens after the application of flexural test

Results

The data of flexural strength were tested for normality using Shapiro-Wilk test, showing not normally distributed data ($p \leq 0.05$). Descriptive statistics of flexural strength was calculated showing the reduction in flexural strength in testing groups from 97MPa up to 75.5MPa, as seen in table 1. Kruskal-Wallis test was used to compare between the groups and showed that all groups were not significantly different from control group, except 15% ($p=0.03$). Flexural strength dropped after addition of micro-glass beads which can clearly be seen in figure 7. The data of Vickers hardness were also tested for normality showing normally distributed data ($p=0.8$). Descriptive statistics of hardness was calculated showing a fluctuation of values around 16 MPa for control group up and down after the addition of fillers, table 2. One-way ANOVA test was used to compare the groups. There was a change in the hardness after micro-glass addition but all changes were not statistically significant ($p=0.2$). The data of test groups were plotted in a box plot, figure 8. Regarding surface roughness test, data were not normally distributed using Shapiro-Wilk test ($p \leq 0.05$). Descriptive statistics of surface roughness was calculated showing an increase in surface roughness following the addition of fillers from 0.53 µm up to 0.77 µm (table 3). Kruskal-Wallis test was used to compare the groups. Surface roughness has increased in test groups; however, this increase was not significantly different from control group ($p=0.11$). These groups were represented as box plot in figure 9.

[DOI: 10.22088/jbums.27.1.18]

Table 1. Descriptive statistics of flexural strength

Test groups	Sample size	Median (MPa) (IQR)	Maximum value	Minimum value
Control	6	97.0 (79-100)[a]	105	41
5%	6	79.5 (67.7-81.7)[a]	87	67
10%	6	81.5 (79-87)[a]	88	79
15%	6	75.5 (66.2-78.7)[b]	84	61

[*]The similar superscript letters refer to insignificant difference, while different letters refer to significant difference between control and test group (Kruskal-Wallis test, p≤0.05)

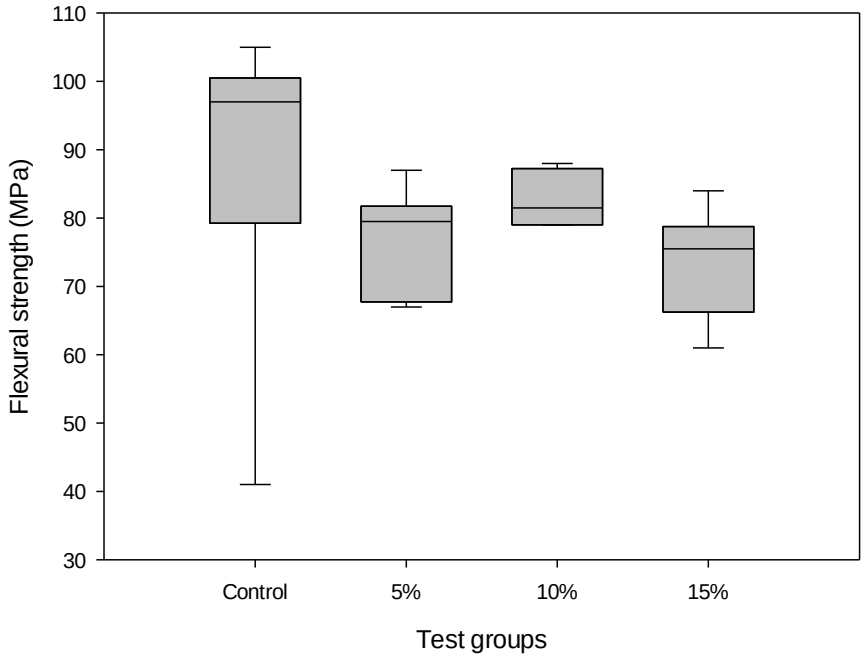

Figure 7. Box plot of flexural strength test groups

Table 2. Descriptive statistics of Vickers hardness

Test Groups	Sample size	Mean (MPa)±SD	Maximum value	Minimum value
Control	6	16.0±1.0[a]	17.6	14.7
5%	6	15.2±1.5[a]	16.5	12.2
10%	6	16.7±0.8[a]	18.0	15.8
15%	6	16.3±1.6[a]	19.2	14.9

[*]The similar superscript letters refer to insignificant difference, while different letters refer to significant difference between control and test group (One-way ANOVA test, p≤0.05)

[DOI: 10.22088/jbums.27.1.18]

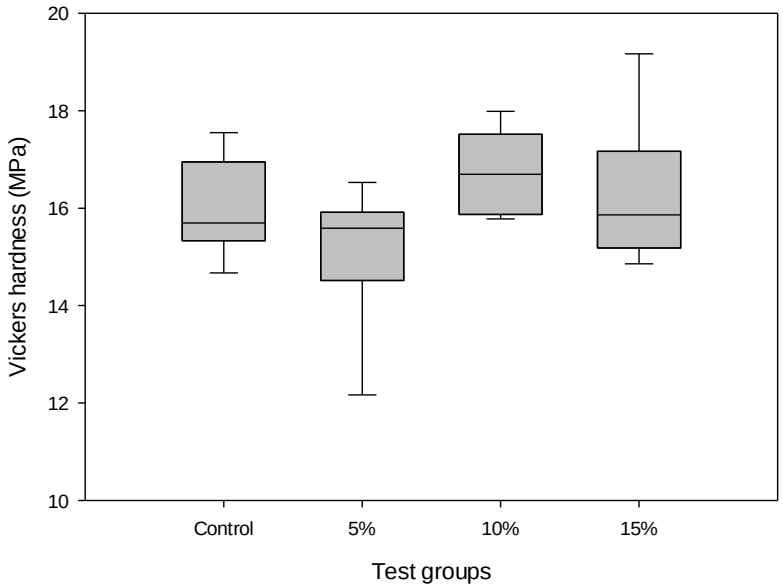

Figure 8. Box plot of Vickers hardness test groups

Table 3. Descriptive statistics of surface roughness (Ra)

Test groups	Sample size	Median Ra (µm) (IQR)	Maximum value	Minimum value
Control	6	0.53 (0.47-0.67)[a]	0.67	0.47
5%	6	0.57 (0.46-0.64)[a]	0.67	0.43
10%	6	0.55 (0.42-0.84)[a]	1.37	0.37
15%	6	0.77 (0.67-0.95)[a]	1.5	0.47

[*]**The similar superscript letters refer to insignificant difference, while different letters refer to significant difference between control and test group (Kruskal-Wallis test, p≤0.05)**

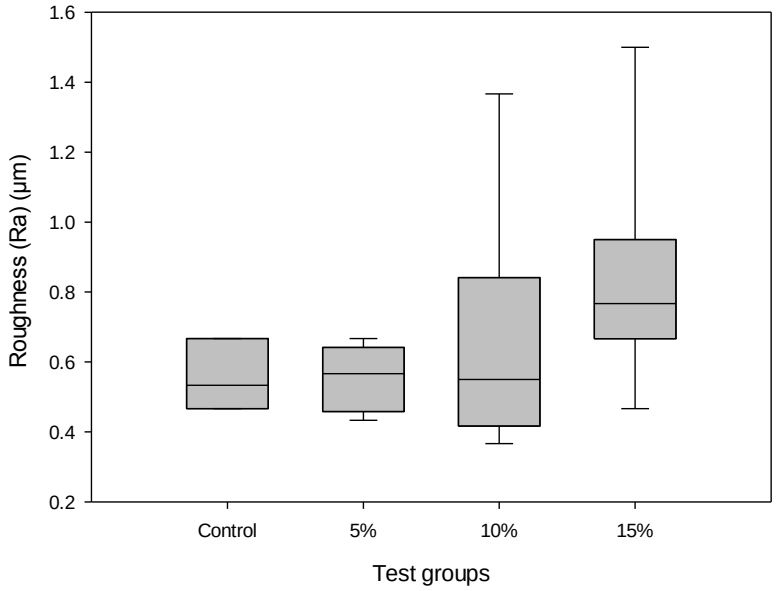

Figure 9. Box plot of surface roughness test groups

[DOI: 10.22088/jbums.27.1.18]

Discussion

According to the results obtained, null hypothesis (1) was rejected, while (2) was accepted. Flexural strength of polymerized Weropress has been adversely affected following the incorporation of micro-glass beads into the powder especially at ratio 15%. Silicon dioxide or silica forms the main constituent of glass beads as mentioned before. According to the previous studies, the addition of silica to polymers increases the flexural strength and hardness (7, 16-18) which is in contradiction with this study. However, some articles come in agreement with the results of this study (20-23). The reduction in strength in these articles and in this study might be due to using unsilanized filler, since silica silanization improves binding with polymer chains (24). On the other hand, some studies showed that this silanization does not produce a remarkable change in strength (23). The other possible reason for that reduction in strength may be due to different method of silica incorporation or different ratios, but some studies showed no considerable difference between different methods of incorporation or different ratios (23). Balos et al. concluded that low ratios of silica dioxide increase flexural properties (25). This is because of insufficient dispersion and aggregation of the high ratio filler in the auto-polymerising acrylic matrix (26). This belief was approved by Alzayyat et al. who found that a ratio of 0.05% of silica is suitable to increase flexural strength (17), while da Silva et al. found that any ratios below 5% silica can increase the strength (18). Most of studies limited the use of reinforcing filler up to 5% (27). Gradual reduction in strength in this study by increasing the ratio until the occurrence of a noticeable difference could be a confirmation of the latter belief.

The hardness values were fluctuating up and down after the addition of fillers with no major change at every ratio. The addition of silica or glass beads usually increases the acrylic hardness with increasing ratios (16), or reducing hardness in some other studies with increasing ratio (25). This difference might be due to the difference in the method of filler incorporation. The former researchers have added fillers to the powder while the latter researchers have added fillers to liquid. However, Mussatto et al. showed no major difference between different ratios, different incorporation methods, and silanization with control. The reason of fluctuation of hardness values could be due to non-homogenous distribution of beads (23).

The roughness values (Ra) increase by the addition of fillers but not noticeably in comparison with control group. This result was in agreement with Mussatto et al. as non-silanized glass beads did not get the enough time to disperse in a quick set auto-polymerising Weropress and the aggregation of fillers within the matrix has increased the surface roughness (23). This is why the increase in roughness was ascending with increasing ratio. Similar results were obtained by Al-Bakri et al. using AFM (28). On the other hand, Cevik et al. disagree with the result of the current study. They found that increasing the silanized silica ratio reduces the roughness (20). The reason might be that increasing filler ratio means increasing silane ratio and further increasing binding and dispersion in a hot cure matric and prohibit of silica agglomeration (29).

The limitations of this study include using certain size of glass beads, mixed with powder only with no silanization, and at a definite ratio. Further suggestions for this research include using different types and sizes of fillers with silanization. Less ratios of fillers are also recommended to be tried in future research starting from 5% and less.

Regarding the limitations of the current study, it was found that the incorporation of micro-glass beads into Weropress powder at 5%, 10%, or 15% by weight showed no strengthening effect on the flexural strength and Vickers hardness, and no noticeable effect on surface roughness of the final product.

[DOI: 10.22088/jbums.27.1.18]

Acknowledgment

We would like to thank the head of department of material science in the University of Technology in Baghdad City “Assis. Prof. Dr Hussein Alaa Jaber” for providing 30% discount on the prices of testing.

[DOI: 10.22088/jbums.27.1.18]

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