

The Effect of Polyester Micro-Particles Powder Addition on Some Mechanical Properties of Light Cured Denture Base Material

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Abstract

In order to improve the light cured denture base material mechanical and physical properties many studies have been conducted by adding different forms and types of filler with various particles size, the aims of the present study was to improve the mechanical properties of light cured denture base material by adding polyester micro-filler particles Two hundred fifty samples were prepared and subdivided according to the percentage of polyester micro-filler particle powder added (0% (Control), 0.5%, 1%, 1.5%, 2%). The mechanical properties tested were Transverse strength, Impact strength, Tensile strength, Surface hardness and Surface roughness (n=10). XRD, EDS, FTIR spectrum and SEM were used to analyze micro-filler Particles distribution and identification of elements in the matrix. The statistical analysis conducted by running one-way ANOVA Table with LSD to show the least significant differences among the groups at P value<0.05. The results show that the highest mean values for transverse strength, tensile strength and surface hardness test found in 2% by weight polyester micro-fillers particles while there were no significant difference reached among all groups in both impact strength and surface roughness when compared with the control group, Within the limitation of this study, the addition of 2% polyester micro-fillers to Light cured denture base material led to improvement in transverse and tensile strength significantly while no significant improvement in impact strength, hardness and changed in surface roughness was noticed.

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Introduction

Polymeric denture base materials are widely used in dentistry, since they were introduced in 1937, dental polymers has a wide use in dental applications due to its relatively inexpensive and easy to process in addition to that it has a superior physical and chemical characteristics such as chemical inert nature and high strength.^{1,2}

Acrylic plastic has been the most widely used denture base material; almost 95% of the plastics in prosthodontics are polymethylmethacrylate

(PMMA).³ Light activated polymers in dentistry are not new, however; the commercialization of Photo polymerization technology started at late 1960s.⁴⁻⁶ In 1984, visible light – cured base resins became available to profession and was suitable for many prosthodontic applications, removable, fixed and maxillofacial prosthesis.⁷⁻⁹

Light cured denture base material (LCDBM), based on urethane dimethacrylate emerge as one of the optional material in removable prosthesis rather than the conventional PMMA because of their synthetic nature that devoted from monomethacrylates.^{10,11}

The interest in LCDBM has been increased due to its superior strength, ease of fabrication, ease of manipulation, complete polymerization without residual compounds that reduce the possibility of hypersensitivity reaction because of the absence of free methylmethacrylate monomer, low exothermic heat, patient's

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preference, and possibility to bond to another resin. Additionally, elimination of traditional mixing, flasking, deflasking and finishing procedure.¹²⁻¹⁵

Ogle et al., in 1986 found that the LCDBM was not toxic, biocompatible, have a higher modulus of elasticity (greater stiffness) than either heat-cured or self-cured acrylic resin.¹⁴ Similar results were obtained by Khan et al., in 1987, Al-Mulla et al., in 1988 and Lewinstein et al., in 1995.^{13,4,16} However, Smith et al., in 1991 and Bouschlicher et al., in 2004 also found that the LCDBM appeared to be harder and more rigid in comparison with other conventional denture base materials but with inherent brittleness and low impact resistance.^{17,18} Moreover, Akin et al., in 2015 concluded that LCDBM possess comparable cytotoxicity to the conventional PMMA with less water sorption when kept in water for long time.¹⁹

Ishigami et al., in 1986 showed that LCDBM has inferior mechanical properties as compared to PMMA denture base material., Al-wahab et al., in 2012 reported that tensile strength of visible light cured acrylic was lower than the heat cured acrylic denture base material while the work of Al-Mulla et al., in 1988 reported that the impact strength of visible light cured acrylic was lower than the heat cured acrylic on the contrary of the hardness values which was higher for the visible light cured acrylic than the heat cured one. Additionally, Aziz in 2015 reported that roughness of visible light cured acrylic was higher than the heat cured acrylic denture base material.^{4,20-22}

AL- Taie and khamas in 2009 found that the transverse strength of repaired LCDBM was inferior when compared to Ivomat pressured cold cured resin and conventional heat cured resin.²³ In addition, Khan et al., in 1988 demonstrated that both visible light-cured and heat-cured resin have comparable values for transverse strength, while Goto *et al.*, in 1986 also investigated the transverse strength of visible light-cured resin and reported that it had a lower value than that of heat-cured resin. This is supported by the work of Andreopoulos et al., in 1991 and Dixon in 1991.^{7,24-26}

Unfortunately, little experimental work had been reported about the physical and mechanical properties of visible light activated resins as denture base materials after the addition of fillers.²⁷⁻²⁹

The idea of reinforcement is not new. Hence; various types of fillers that can be added to acrylic resins to improve their physical and mechanical properties has been available. Reinforcement provides strength and rigidity, helping to support structural load. The position and direction of the reinforcement can maintains the binder or matrix (inorganic or organic). Significantly, constituents of the composites retain their specific chemical and physical properties.^{30,31}

Dixon et al., in 1992²⁶ reported that the incorporation of polyethylene fibers to visible light cure denture base material cause significant increase in transverse strength. The study of Ali in 2017³² suggested that Addition of SiO₂ nanofillers to (LCDBM), slightly improve the transverse strength and surface hardness while the surface roughness shows non-significant increment.

The goal of the present study is to examine different properties of LCDBM such as transverse strength, impact strength, tensile strength, hardness and surface roughness after addition of different concentrations of polyester (Polyethylene terephthalate) micro-particles powder.

Materials and methods

Polyester micro- filler particles powder with average particle size of 300 micron (Goodfellow Cambridge limited, England) was added to the liquid of the tray LCDBM (Aurora VLC) in a concentration of (0.5%, 1%, 1.5%, 2%) by weight respectively, while 0% represents control group. 250 specimens were prepared. The specimens were divided into 5 groups according to the test utilized in the study. Fifty samples were selected for each test (10 samples for the control and the other 40 samples were assigned for different concentration of polyester micro-particles powder groups) (n=10).

The powder of the tray LCDBM were weighed by electronic balance and the percentage of polyester fillers were subtracted from the powder and added to the liquid according to groups included in the study, the polyester fillers mixed with the liquid in a jar by a special mixer for 5 min. and then added to the powder to complete the mixing procedure.

Two types of mold were made, the first one has 3 rectangular holes (80mmx10mmx4mm);

length, width and thickness respectively, prepared by CNC machine (Computer numerical control) for impact strength while the second mold has also 3 rectangular holes (65mmx10mmx2.5mm), for tensile strength, transverse strength, shore D surface hardness and surface roughness. After coating the mold with Vaseline, the lower transparent cover was also coated with Vaseline and reassembled with the middle part, then the material was adapted carefully in the mold and excess material removed by sharp knife; then the upper cover placed on the mold after coated with a layer of Vaseline and tightened with screw and nuts so that the middle part is sandwiched between the two transparent covers. The mold was transferred into the light cure chamber (KT-888, China, Wavelength of 400 μm) so that the upper surface left for 7.5 min, then the mold was inverted and cured again for another 7.5 min. (total curing time 15min.). When curing was completed the pattern were removed carefully, then finished and polished except for samples of surface roughness.³²

Testing procedure:

Transverse strength test. The specimens were fabricated with dimensions of (65mmx10mmx2.5mm), length, width and thickness respectively. After conditioning in water at 37°C for 48 hrs.³³, the samples were subjected to a 3 point bending test using an Instron machine (Laryee CO., Ltd. China). The stress was recorded and determination of the transverse strength follows this formula:

$$\text{Transverse strength} = 3Pl/2bd^2 \text{.}^{34}$$

Where:

P: is the peak load

l: is the span length

b: is the sample width

d: is the sample thickness.

Impact strength test. Specimens were kept in distilled water for 48 hrs. at 37°C before being tested. Rectangular samples of dimension (80mmx10mmx4mm), were fabricated. The test was performed using Izod impact testing machine (Time testing machine, China).³⁵ The energy absorbed by the un-notched specimens was calculated using this equation:

$$\text{Impact strength} = (E/bd) \times 10^3 \text{.}^{34}$$

Where:

E: is the impact energy absorbed in joules.

b: is the width of the sample.

d: is the thickness of the sample

Tensile strength test. The samples were prepared with dimension of (65mmx10mmx2.5mm) and stored in distilled water for 48hrs at 37°C before being tested by an instron testing machine. The machine induced force that resists the elongation of a material in a direction parallel to the direction of the stress at a cross head speed of (1 mm/min), the load was measured by a tensile load cell with a maximum load of (100kg).^{33,34}

Specimens were loaded until fracture and the load of fracture was recorded from the instron graph reader in Kilograms (kg) which were converted into Newton (N).³⁶

The value of tensile strength was calculated by the followed formula:

$$\text{Tensile strength} = (F/A)$$

Where:

F= Force at failure (N)

A= Area of cross section at failure (mm^2).

Shore D surface hardness. The samples were prepared with dimension of (65mmx10mmx2.5mm), and stored in distilled water for 48hrs. at 37°C.³³ Shore D hardness tester (Elcometer, Germany) with a calibrated scale from (0-100 units) was used. The final value of this test was obtained by calculating the average value of 5 readings performed for each sample. All measurements were done by one person.

Surface roughness. The surface roughness samples of (LCDBM) were measured by Profilometer device (TR200, Time CO. China). The samples utilized in this test were prepared with dimension of (65mmx10mmx2.5mm). Later on, the samples kept in distilled water for 48 hrs. at 37°C,³³ then the test was performed by Profilometer apparatus that capture surface irregularities. Three readings for each sample were determined and the average value of these readings represents the final value.

Results

X-ray diffraction (XRD) analysis. Figure 1 show the XRD spectral results for all groups, there was a change in the spectrum between 20 and 40 \AA degree in group with polyester micro-

particles powder added to LCDBM as compared to other spectrum for the other groups.

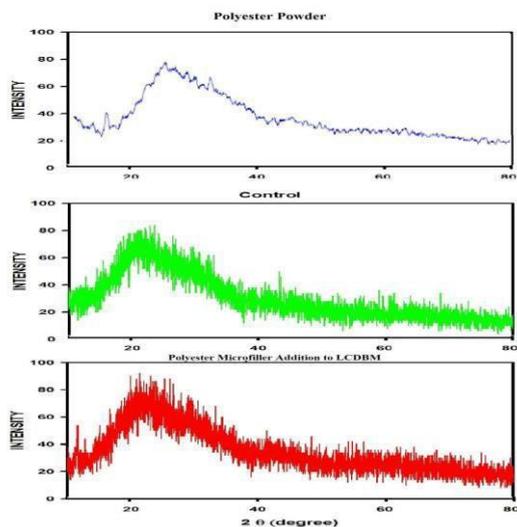


Figure 1: XRD spectrum for polyester filler and LCDBM before and after the addition of polyester micro-fillers

Energy dispersive spectroscopy (EDS) Analysis. It shows that there were noticeable change in the elements distribution and percentage of the trace elements found within the matrix analyzed as the control group compared with the groups containing polyester micro-particles (fig. 2).

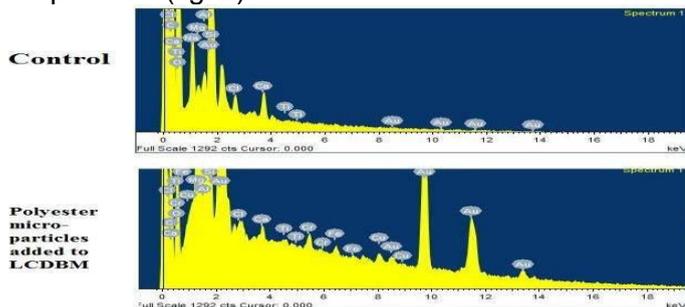


Figure 2: EDS Analysis.

FTIR Analysis. Figure 3 shows that a change in the FTIR spectrum observed in the range 2350 -cm in which a (C=N) stretch bond observed when compared with the FTIR spectrum of the control group.

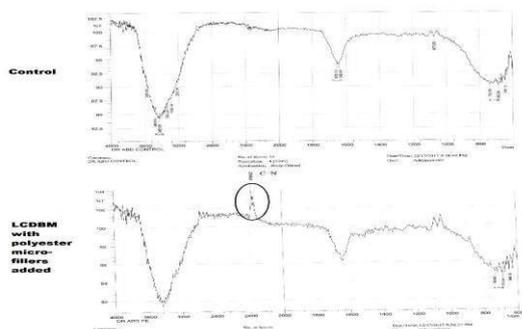


Figure 3: FTIR Analysis.

Scanning electron microscope (SEM) examination. Figure 4 shows the SEM results for control and LCDBM with polyester micro-particles added, there are some voids and pores appeared in addition to the polyester micro-particles dispersed randomly throughout the specimen.

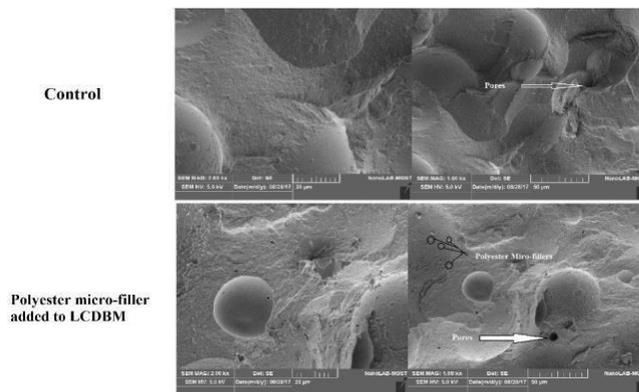


Figure 4: Scanning electron microscope analysis Mechanical Properties.

Figure 5 represent bar chart plot for the mean of all groups for all tests.

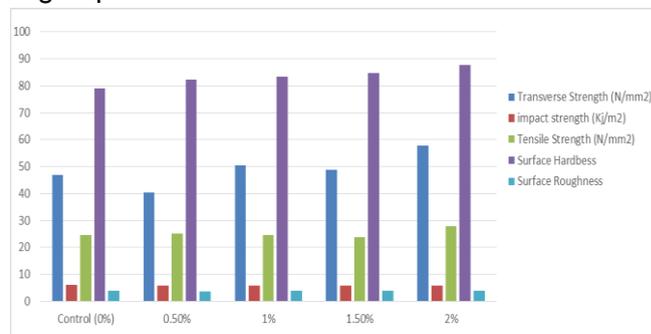


Figure 5: Bar chart representation for the mean of all groups for all tests

Transverse strength test. The descriptive analysis, one way analysis of variance (ANOVA) and post hoc LSD test of the Transverse strength is summarized in (Tables 1 and 2). One way ANOVA indicated highly significant difference between the experimental groups when comparing the mean values for transverse strength test.

% of fillers	N	Mean (N/mm ²)	Std.	Min.	Max.	F-test	P. value	Sig.
Control (0%)	10	46.96	1.734	45	49.4	106.317	0.000	HS
0.5%	10	40.45	1.211	39.4	42.6			
1%	10	50.346	1.699	48.6	53.03			
1.5%	10	48.92	2.883	44.7	52.5			
2%	10	57.76	1.624	55.4	60.1			

Table 1. Descriptive analysis and one way ANOVA table for transverse strength test.

Post hoc LSD test results showed there were no significant differences between (1% by weight

polyester micro-filler group) and (1.5% by weight polyester micro-filler group), in addition, there was significant differences between (0% by weight polyester micro-filler group) and (1.5% by weight polyester micro-filler group). Moreover, highly significant differences were found between the rests of the experimental groups.

% of fillers		Mean difference	P. value	Sig
0%	0.5%	6.51	0.000	HS
	1%	- 3.386	0.000	HS
	1.5%	- 1.96	0.027	S
	2%	- 10.8	0.000	HS
0.5%	1%	- 9.896	0.000	HS
	1.5%	- 8047	0.000	HS
	2%	- 17.31	0.000	HS
1%	1.5%	1.426	0.103	NS
	2%	- 7.414	0.000	HS
1.5%	2%	- 8.84	0.000	HS

Table 2. Post hoc LSD multiple comparison for transverse strength test. p value \geq 0.05 (Non- significant), p value $<$ 0.05 (Significant), p value $<$ 0.01 (High significant).

Impact strength test. The descriptive analysis, ANOVA Table and post hoc LSD test of the Impact strength test is summarized in Tables 3. One way ANOVA indicated no significant difference among the experimental groups when comparing the mean values for the impact strength test.

% of fillers	N	Mean (Kj/m ²)	Std.	Min.	Max.	F-test	P. value	Sig.
0%	10	6.07	0.385	5.75	6.75	1.052	0.391	NS
0.5%	10	5.88	0.097	5.75	6			
1%	10	5.92	0.063	5.85	6			
1.5%	10	5.92	0.201	5.65	6.2			
2%	10	5.95	0.218	5.65	6.25			

Table 3. Descriptive analysis and one way ANOVA Table for impact strength test.

Tensile strength test. The descriptive analysis, ANOVA Table and post hoc LSD test of the Tensile strength test is summarized in Tables 4 and 5. One way ANOVA Table indicated highly significant difference among the experimental groups in the tensile strength test. Post hoc LSD test results showed there were no significant differences between 0% by weight polyester micro-filler group and 0.5%, 1% and 1.5% by weight polyester micro-filler group, also no significant difference was detected between 1% by weight polyester micro-filler group and 0.5% and 1.5% by weight polyester micro-filler group.

In addition, there was significant differences between 0.5% by weight polyester micro-filler group and 1.5% by weight polyester micro-filler group. Moreover, highly significant differences was found between 2% by weight polyester micro-filler group and 0%, 0.5%, 1% and 1.5% by weight polyester micro-filler group.

% of fillers	N	Mean (N/mm ²)	Std.	Min	Max	F-test	P. value	Sig
0%	10	24.7	2.477	20.2	27.1	12.622	0.000	HS
0.5%	10	25.22	0.9	24.2	26.2			
1%	10	24.62	0.654	23.6	25.2			
1.5%	10	23.64	1.251	22.4	25.4			
2%	10	27.84	0.974	26.4	29			

Table 4. Descriptive analysis and one way ANOVA Table for tensile strength test.

% of fillers		Mean difference	P. value	Sig
0%	0.5%	- 0.52	0.413	NS
	1%	0.08	0.899	NS
	1.5%	1.06	0.099	NS
	2%	- 3.14	0.000	HS
0.5%	1%	0.6	0.345	NS
	1.5%	1.58	0.016	S
	2%	- 2.62	0.000	HS
1%	1.5%	0.98	0.126	NS
	2%	- 3.22	0.000	HS
1.5%	2%	- 4.2	0.000	HS

Table 5. Post hoc LSD multiple comparison for tensile strength test. p value \geq 0.05 (Non- significant), p value $<$ 0.05 (Significant), p value $<$ 0.01 (High significant).

Shore D hardness test. The descriptive analysis, one way ANOVA Table and post hoc LSD test of the Hardness strength test were summarized in Tables 6 and 7. One way ANOVA Table indicated highly significant difference among the experimental groups when comparing the shore D surface hardness mean values.

Post hoc LSD test results showed there were highly significant differences among the experimental groups except for no significant differences between 0.5% by weight and 1.5%, and 1% by weight and 2% by weight polyester micro-filler group.

% of fillers	N	Mean	Std.	SE	Min.	Max.	F-test	P. value	Sig.
0%	10	78.96	2.789	0.882	75	81.7	32.655	0.000	HS
0.5%	10	82.26	0.79	0.249	81.5	83.4			
1%	10	83.3	1.9	0.602	80	85			
1.5%	10	84.62	1.151	0.364	82.8	85.8			
2%	10	87.66	1.481	0.468	85.5	89.3			

Table 6. Descriptive analysis and one-way Analysis of variance (ANOVA) for Surface hardness test.

% of fillers	Mean difference	P. value	Sig
0%	0.5%	-3.3	.000 HS
	1%	-4.34	.000 HS
	1.5%	-5.66	.000 HS
	2%	-8.7	.000 HS
0.5%	1%	3.3	.000 HS
	1.5%	-1.04	.194 NS
	2%	-2.36	.004 HS
1%	1.5%	4.34	.000 HS
	2%	1.04	.194 NS
1.5%	2%	5.66	.000 HS

Table 7. Post hoc LSD multiple comparison for Hardness test, p value < 0.05 (Non-Significant)

Surface roughness test. The descriptive analysis, ANOVA Table and post hoc LSD test of the surface roughness test is summarized in Tables 8. One way ANOVA Table indicated no significant difference among the experimental groups when comparing the surface roughness mean values.

% of fillers	N	Mean	Std.	Min.	Max.	F-test	P. value	Sig.
0%	10	3.812	0.176	3.65	4.1	0.829	0.514	NS
0.5%	10	3.76	0.139	3.59	3.95			
1%	10	3.862	0.093	3.7	3.97			
1.5%	10	3.792	0.136	3.66	3.99			
2%	10	3.786	0.099	3.67	3.9			

Table 8. Descriptive analysis and one way ANOVA Table for surface roughness test.

Discussion

In spite of the acceptance of LCDBM as a restorative material in dentistry, this material exhibit weak mechanical properties especially

flexural and impact strength.³² Additions of different concentrations of polyester filler can be considered as an attempt to improve some of these material properties.

The resistance of the material to breakage (stiffness) should be studied to evaluate its performance during mastication; this can be done by measuring the transverse (flexural) strength. Transverse strength is the force needed to deform the material to fracture or irreversible yield.^{37,38} It is a combination of compressive, tensile and shear strengths, all of which directly reflect the resistance and stiffness needed to fracture the material.^{29,39}

The results of the present study shows improvement in transverse strength as the percentage of micro-fillers increased, thus, LCDBM reinforced with 2% polyester micro-fillers shows highest transverse strength values. This might be due to the dispersion of the fillers into the polymer matrix aiding in the continuity of the resin matrix by filling interstitial spaces. Therefore, the shear strength between polyester filler and urethane dimethacrylate polymer chain will be enhanced.^{40,41} The results of this study are in agreement with the finding of Qasim et al., in 2012,³⁵ who found that the mean of flexural strength of LCDBM was improved when carbon nanotubes were added and Ali in 2017 who reported slight improvement in transverse strength of LCDBM reinforced with 2% SiO₂ nanofillers.³²

Impact strength is the ability of a material to resist a sudden high level force or shock.^{37,38} For impact strength, sample without notches were chosen rather than notched one, since it was difficult to fabricated, time consuming, and weakened the sample. In addition to that, material brittleness raise a difficulty in notch preparation.³² The results of the present study show increase in the impact strength as percentage of fillers increased but still less than the control group, hence; LCDBM reinforced with 2% polyester fillers reduce impact strength values slightly. Possibly because the urethane dimethacrylate stiffness attained by filler addition unfortunately results in more stresses inside the resin matrix especially adjacent to the borders of polyester filler which might enhance fracture by sudden impact.⁴² In fact, the brittle nature of the

material and aggregation of polyester micro-particles inside some voids or pores, which might resulted from polymerization or evaporation of the liquid, could facilitate the formation of micro-cracks within the urethane matrix which would weakened the impact strength as revealed by SEM results. The results of this study are in agreement with the finding of Ali in 2017 who reported reduction in the impact strength values of LCDBM reinforced with 2% SiO₂ nanofillers and disagreed with Qasim et al., in 2012 who reported increase in impact of LCDBM when carbon nanotubes were added. This could be due to different measuring device, fillers and material.^{32,35}

In practice, a good equilibrium between flexural and impact strength is required to have a rigid mixture with improved impact strength is needed. Although such a balance is important but positive correlation between such parameter is not always possible in practice. Sometimes one enhanced and the other properties are deteriorated.⁴³

Another mechanical properties have been studied which is the tensile strength. The reason behind choosing this material properties to be studied is that the material much weaker in tension than in compression, which may contribute to failure of the material in service.⁴⁴ The results of the present study show minimum decrease in the tensile strength in 1% and 1.5% polyester fillers as compared with the control group, while slight increase in 0.5% filler is seen. The 2% of fillers show the highest tensile strength values, hence; LCDBM reinforced with 2% polyester fillers cause increase the tensile strength values. This may be due to action of the micro fillers particles that intend to fill the gaps and pores between the matrixes of the material that lead to enhance the molecular attraction and hence increase the tensile strength of the bulk materials.

Hardness is term used to describe the resistance of the material to indentation and also it is a measure of the resistance to wear or scratching. The surface hardness of the experimental samples was increased in 0.5% group d again. The 2% group shows hardness value almost resembling the control group. This increase in hardness values may possibly

because the modulus of elasticity and polymerization shrinkage were improved when micro particles were added. Moreover, such improvement might be due to fair distribution of polyester filler in polymer matrix which limit deformation under load.⁴⁵

The surface roughness properties of any denture base material is of a specific concern as studies of denture base materials have indicates a direct link between this feature and the plaque retention and the adherence of *Candida Albicans*. The result of this study reported non-significant decrease in surface roughness of the experimental specimens, this may be due to the nature of the polyester micro-particles added and its physical properties that may reduce the surface roughness.

Conclusions

This in vitro study showed that LCDBM reinforced with 2% polyester micro filler significantly improve the transverse strength and tensile strength. However, this addition lowered the impact strength, shore D surface hardness and surface roughness non significantly, suggests further laboratory studies with other fillers or modification in mixing technique to achieve higher improvement and better balance between flexural and impact strength.

Declaration of interest

The author report no conflict of interest and the article is not funded or supported by any research grant.

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