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ORIGINAL ARTICLE

EFFECT OF IMMERSION AND INCORPORATION OF POLYVINYL CHLORIDE ON THE FLEXURAL STRENGTH AND MICRO HARDNESS OF POLYMETHYLMETHACRYLATE DENTURE BASE RESIN

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ABSTRACT

OBJECTIVE: To find the effect of artificial saliva, water and different percentages of polyvinyl chloride (PVC) on the flexural strength and microhardness of heat cure polymethylmethacrylate (HC-PMMA) denture base resin.

METHODS: An in-vitro study conducted, from April-December, 2020, at the MRL University of Peshawar and Interdisciplinary Research Centre in Biomedical Materials (COMSATS) Lahore. Total of 100 specimens for five groups (n=20) denoted by A, B, C, D and E containing 0%, 5%, 10%, 15% and 20% PVC in HC-PMMA respectively were fabricated to test flexural strength. Each group was divided into two subgroups depending on immersion medium containing ten specimens each. Three-point bending test was used to determine flexural strength. Microhardness was determined using thirty (n=30) specimens through Vickers hardness tester. Statistical analysis was performed by using one way ANOVA and post hoc Tukey test. P-value <0.05 was considered as significant.

RESULTS: After immersion in water, one way ANOVA showed significant effect on flexural strength and Post-hoc Tukey's test showed significant difference between group C and E ($p<0.001$) and, D and E ($p< 0.001$). Microhardness of A was significantly different from E after immersion in water ($p<.05$). Similarly, after immersion in artificial saliva, one way ANOVA showed statistically significant effect on microhardness ($p< 0.01$) and Post-hoc Tukey's test showed significant difference between group C and E ($p<0.01$) and, A and C ($p<0.01$).

CONCLUSION: Flexural strength of HC-PMMA impregnated with 20% PVC increased after immersion in water, similarly, HC-PMMA containing 10% PVC shows increased microhardness after immersion in artificial saliva.

KEYWORDS: Flexural Strength (MeSH); Acrylic Resins (MeSH); Acrylic denture (Non-MeSH); Heat cure polymethylmethacrylate (Non-MeSH); Polymethyl Methacrylate (MeSH); Polyvinyl Chloride (MeSH); Hardness (MeSH); Hardness Tests (MeSH); Dental Bases (MeSH)

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INTRODUCTION

Heat cure polymethylmethacrylate (HC-PMMA) is most widely used denture base material for fabricating removable partial and complete dentures.[1] It is a colorless, biocompatible and thermoplastic material. Despite of frequent use, the fracture of denture base occurs due to low flexural and fatigue strengths.[2] In one survey, 70% acrylic resin base dentures were found cracked in their initial three years of insertions.[3] Acrylic denture base can be cracked while subjected to excessive chewing forces or due to even minor accidents. This eventually leads to reduced clinical life of the prostheses, and therefore increases the patient's dental visits and the cost of the treatment.[3] Therefore, to avoid fracture of the dentures, the thickness of acrylic resin in prone regions was increased or reinforced. Copolymerization by rubber with HC-PMMA, reinforcement by metallic wire, fibers and the use of oxides of metal were tried to increase the properties of HC-PMMA denture base material.[4]

Excessive biting and chewing forces can distort the acrylic denture base during normal functions as it can change the stress distributions and cause denture base fracture.[5] Frequently used polymerization techniques for HC-PMMA denture bases are water bath and microwave polymerization.6 During laboratory workup of HC-PMMA, different surface flaws like pores and discrepancies can be developed through which fungi and bacteria can stick and decrease the elastic modulus and flexural strength.[6]

Unplasticized polyvinyl chloride is a transparent thermoplastic material, which has manufacturing, and engineering applications due to its toughness, inertness, flexibility and low environmental erosion. It is inexpensive, colorless material and possesses good mechanical and insulating properties due to high polar nature. PVC/HC-PMMA blend is miscible due to the existence of a specific intermolecular interaction between PVC and HC-PMMA.[7] Researchers have investigated different properties of either PVC or HC-PMMA or both of this polymeric system using different concentration of PVC incorporated in HC-PMMA.[7-9]

Aouachria, et al. investigated the properties of plasticized PVC/HC-PMMA blends using variable composition of HC-PMMA/PVC blend for dynamic thermo gravimetric analysis and thermo-oxidative degradation.[10] They concluded that an increased quantity of the HC-PMMA in the blend improved the thermoxidative stability of PVC and HC-PMMA blend.

Jha et al. evaluated effect of gamma irradiation on PVC, HC-PMMA and their

TABLE I: MATERIALS USED IN THE STUDY

Patient Variables	Manufacturer	Batch number
Heat cure polymethylmethacrylate (HC-PMMA)	Mr. Dental Ltd UK	8.09
Polyvinyl chloride (PVC)	Engro Polymers, Pakistan	15.83
Type 3 dental stone	Guangdong China	10.69
Cold mould seal	Ivoclar Vivadent Corporate	

TABLE II: COMPOSITION OF ARTIFICIAL SALIVA[10]

Composition of artificial saliva	Quantity per 1000 g
Sodium chloride (NaCl)	0.400 g
Potassium chloride (KCl)	0.400 g
Calcium chloride, monohydrate ($CaCl_2$ H_2O)	0.795 g
Sodium dihydrogen phosphate (NaH_2PO_4)	0.69 g
Sodium sulfide nonahydrate (Na_2S. $9H_2O$)	0.005 g
Urea	1.00 g
Distilled water	1000 ml

TABLE III: MEAN VALUES AND STANDARD DEVIATION OF FLEXURAL STRENGTH (MPA) OF VARIOUS GROUPS (N=10) STORED IN ARTIFICIAL SALIVA AND WATER FOR 7 DAYS

Groups	Flexural Strength (Mpa)	
	Artificial saliva (Mean±SD)	Water (Mean±SD)
A (Control)	86.04±9.97	86.93±7.92
B (5% PVC)	79.87±7.85	86.88±5.65
C (10% PVC)	79.43±6.40	80.64±9.78
D (15% PVC)	82.48±5.07	79.15±5.55
E (20% PVC)	84.27±10.25	92.06±4.98

PVC: polyvinyl chloride

TABLE IV: ONE-WAY ANOVA OF FLEXURAL STRENGTH OF SPECIMEN'S GROUPS AGED IN ARTIFICIAL SALIVA AND WATER

	Sum of Squares		df		Mean Square		F		Sig.	
	Artificial saliva	Water	Artificial saliva	Water	Artificial saliva	Water	Artificial saliva	Water	Artificial saliva	Water
Between Groups	319.55	1075.806	4	4	79.89	268.952	1.200	5.468	0.324	0.001
Within groups	2995.46	2213.375	45	45	66.56	49.186				
Total	3315.01	3289.182	49	49						

polymer blends with different weight percentages ratio.[8] They reported a significant effect on microhardness of the blend. They also concluded that microhardness of irradiated pure PVC was more than irradiated pure HC-PMMA due to radiational cross linking. He reported a decrease in hardness with increase in content of HC-PMMA in PVC (5 to 20 weight %) at different loads. The decrease in hardness with increase in the content of HC-PMMA is slow at low load (10g), and the rate increases with increase in load. Blend of HC-PMMA and PVC gives an indication of feasibility of producing better grade polymers from polymer blend in term of a cost, toughness and strength.[9] All of these changes may be attributed to the excellent properties of PVC, including its impact strength, flexural strength and low water absorption. However, it remains unknown that whether saliva can influence the mechanical properties of blend and there is scarce literature about the effect of artificial saliva and incorporation of different concentration of PVC/HC-PMMA blend on the mechanical properties of HC-PMMA. Therefore, the aim of this study is to evaluate the effect of immersion and different percentages of unplasticized PVC in HC-PMMA on its flexural strength and microhardness

METHODS

This was an in vitro study conducted, during the period from April, 2020 to December, 2020, at the Material Research Laboratory (MRL) University of Peshawar and Interdisciplinary Research Centre in Biomedical Material (IRCBM), COMSATS, Lahore, Pakistan. The study was approved by the board of advanced study & research of the university as well as its institutional review board. The details about the materials used are given in Table I and II.

Preparation of HC-PMMA/PVC Blend

Rectangular shaped metallic mould having dimension 65 x 10 x 3 mm length, width and thickness respectively was used to prepare test specimens based on ISO standard 1567. Total of hundred specimens were prepared for five groups denoted by A, B, C, D and E containing 0%, 5%, 10%, 15% and 20% of PVC in HC-PMMA respectively. Hundred sample size was based on previous study.[17] Each group was divided into two sub groups having ten specimens each depending on the immersion medium used. The HC-PMMA-PVC blend was mixed using horizontal lab mill (rpm 100–1000). Horizontal ball milling is a two-roller system, in which both rollers are rotating at speed of 100-1000 rpm. A stainless jar was used with stainless steel balls of 10 diameter and 4g weight. Powder of polymethylmethacrylate and PVC were weighed by weighing machine and labelled according to their group name (A, B, C, D, and E). The powder of each group was putted in a stainless jar and put on sliding roller. The time for ball milling machine was 1 hour.[11]

For fabrication of each sample, a wax pattern was made in the metallic mould and was invested in a dental flask with type 3 dental stone. Tapping of flasks were carried out during investment procedure via mechanical vibrator (IRIS, AX-Z^2, Tiajin, China) to prevent air bubble. Dental flask was immersed in boiling water for 10 minutes to remove waxes after complete setting of hard plaster. After opening the flasks, the residual wax was removed by washing with hot water and placed for drying. Cold mold seal was applied on both surfaces of flasks and conventional and

TABLE V: POST HOC TUKEY TEST FOR FLEXURAL STRENGTH OF SPECIMENS IN VARIOUS GROUPS AGED IN ARTIFICIAL SALIVA AND WATER

Groups		*p* - value	
		Artificial saliva	Water
A (Control)	B (5% PVC)	0.45	0.99
	C (10% PVC)	0.38	0.43
	D (15% PVC)	0.86	0.20
	E (20% PVC)	0.99	0.32
B (5% PVC)	C (10% PVC)	1.00	0.29
	D (15% PVC)	0.95	0.12
	E (20% PVC)	0.75	0.47
C (10% PVC)	D (15% PVC)	0.92	0.99
	E (20% PVC)	0.68	0.006
D (15% PVC)	E (20% PVC)	0.99	0.001

PVC: polyvinyl chloride

TABLE VI: MEAN VALUE AND STANDARD DEVIATION OF MICROHARDNESS (VHN) OF VARIOUS GROUPS (N = 06) STORED IN ARTIFICIAL SALIVA AND WATER FOR 7 DAYS

Groups	n	Microhardness (VHN)	
		Artificial saliva (Mean±SD)	Water (Mean±SD)
A (Control)	06	14.73±2.21	18.7867±2.18
B (5% PVC)	06	18.23±3.16	15.6033±3.79
C (10% PVC)	06	22.53±0.86	15.1833±1.81
D (15% PVC	06	18.91±1.25	13.7833±3.14
E (20% PVC)	06	12.82±2.92	11.2867±0.61

PVC: polyvinyl chloride; VHN:Vickers hardness number

TABLE VII: ONE-WAY ANOVA OF MICROHARDNESS OF SPECIMEN'S GROUPS AGED IN ARTIFICIAL SALIVA AND WATER

	Sum of Squares		df		Mean Square		F		Sig.	
	Artificial saliva	Water	Artificial saliva	Water	Artificial saliva	Water	Artificial saliva	Water	Artificial saliva	Water
Between Groups	172.276	89.940	4	4	43.069	22.485	8.360	3.443	0.003	0.051
Within groups	51.519	65.310	10	10	5.152	6.531				
Total	233.795	155.251	14	14						

TABLE VIII: POST HOC TUKEY TEST FOR MICROHARDNESS OF SPECIMENS AGED IN ARTIFICIAL SALIVA AND WATER

Groups		*p* - value	
		Artificial saliva	Water
A (Control)	B (5% PVC)	0.38	0.57
	C (10% PVC)	0.01	0.46
	D (15% PVC)	0.23	0.19
	E (20% PVC)	0.83	0.03
B (5% PVC)	C (10% PVC)	0.21	1.00
	D (15% PVC)	0.99	0.90
	E (20% PVC)	0.09	0.30
C (10% PVC)	D (15% PVC)	0.35	0.95
	E (20% PVC)	0.003	0.39
D (15% PVC)	E (20% PVC)	0.05	0.75

PVC:polyvinyl chloride

reinforced HC-PMMA was inserted into the moulds. The ratio of polymer to monomer was 2.5:1 by weight.[12] Polymer and monomer were mixed by using a cement spatula in a ceramic jar for 30 sec and the jar was covered with a lid to obtain the dough stage of polymerization. The polymerized mass in dough stage was packed in already prepared moulds and pressed with hydraulic press at 200 bars for 5 minutes. The flash were removed with sharp scalpel.[13] Short polymerization cycle was used by immersing the clamped flask in water bath at 74 °C for 1.5 hours and terminal boil for 30 minutes.[14] The specimens were retrieved by opening the flask after cooling at room temperature and finished manually and by using electric motors with different grit sand papers of 400, 600 and 800 grit size. The selected specimens were placed in tap water at room temperature and artificial saliva for 07 days before testing.

Flexural Strength

Flexural strength was determined using three point bend test based on ISO standard: 1567:1999 (Dentistry-denture base polymer) in a universal testing machine (Series LFV-1.5KN). [13,15] Each specimen was mounted in specially designed jig with 50mm fixed distance between two supports and central loading was performed at cross speed of 5 mm/min until the fracture of specimen occurred. The maximum applied force was recorded and the flexural strength (MPa) was calculated by using the formula:[16]

$$F=3WL/2bd^2$$

Whereas W: Maximum force at fracture, F: flexural strength, L: distance between two supports (50 mm), b: Width of specimen (10 mm) d; Thickness of specimen (3 mm).

Microhardness

Microhardness was determined by using the fragments of thirty specimens fractured during flexural strength testing.[17] Each group contained 06 fragments and microhardness was measured by Vickers hardness testing. By using Vickers hardness tester - the dimensions of indentation produced in the test specimen was determined by applying a load of 2.94 N (0.229 kg) with a dwelling time of 15 seconds and 30X magnification at room temperature. For each specimen three indentations were made at different points and mean

hardness value was calculated using formula:

$$VHN = 1.8544\ F/d^2$$

Where F is the load (kg), d is the average between two measured indentation diagonals (mm) and VHN is the Vickers hardness number.

The SPSS version 26 was used for analysis of results by applying analysis of variance (ANOVA) and Tukey 's post hoc test to determine the significance of the difference between the groups. P- value less than 0.05 was considered as significant.

RESULTS

Flexural Strength

The results of the flexural strength determined after immersion in artificial saliva are presented in Table III. Control group A exhibited the highest flexural strength, whereas, in experimental groups the group E showed highest while group C revealed the lowest flexural strength. One-way ANOVA showed statistically insignificant difference among the various groups ($p>0.05$) (Table IV). Post hoc Tukey test showed that group B containing 5% PVC showed decrease in strength ($p>0.05$) while insignificant increase was observed when the percentage of PVC was increased beyond 10% (C) ($p>0.05$), (Table V).

Table III also shows the results of the flexural strength for the groups A, B, C, D and E respectively after immersion in water. Group E showed the maximum while group D showed the minimum flexural strength among the various groups. Flexural strength decreased on increasing the percentage of PVC from 5-15% except group E (20% PVC) which exhibited increase in flexural strength. One-way ANOVA showed statistically significant difference among the various groups ($p <0.05$) (Table IV). Post hoc Tukey's test showed that the flexural strength of group E was significantly different when compared to group D ($p<0.05$) while the rest of the groups showed statistically insignificant differences when compared to each other ($p>0.05$) (Table V).

Microhardness

Table VI shows the results of the microhardness for the various groups after immersion in artificial saliva. Group C showed the highest microhardness while group E showed the lowest microhardness among the experimental groups. Microhardness increases with increasing the percentage of PVC from 5–10% except group E (20%) which exhibited decrease in microhardness. One-way ANOVA showed statistically significant difference among the various groups ($p<0.05$) (Table VII). Post hoc Tukey's test showed that the microhardness of group E was significantly different when compared to group C and D ($p<0.05$), similarly group C showed significant difference when compared to group A while the rest of the groups showed statistically insignificant differences when compared to each other (Table VIII).

Table VI also shows the results of the microhardness for the various groups after immersion in water. Group A (control) exhibited the highest microhardness while group E showed the lowest microhardness among the experimental groups. Microhardness decreases with increasing percentage of PVC concentration. One-way ANOVA indicated statistically insignificant difference among the various groups ($p>0.05$) (Table VII). Post hoc Tukey's test showed that the microhardness of group C was statistically significant when compared to group A, similarly microhardness of group E was significantly different when compared to group C ($P<0.05$) (Table VIII).

DISCUSSION

Fracture in denture base is most commonly encountered clinical problem in prosthodontics, therefore various studies were conducted to improve the mechanical properties of denture base resin.[18] In the present study, the flexural strength of HC-PMMA impregnated with various weight percentage of PVC was insignificantly decreased after immersion in artificial saliva. Viebke & Gedde stated that chlorine compounds have strong oxidation potential and water containing chlorine usually affect the mechanical properties of PVC, for this reason strong stabilizers are added for plasticizing effect.[19] In present study, unplasticized PVC was used therefore it might be the reason that the flexural strength was decreased with increasing percentage of PVC, which is in accordance to previous study.[20] The flexural strength might be decreased due the PVC particles that may acted as impurities to conventional HC-PMMA that increased the plasticization and resulted in decrease in flexural strength.[21] In contrary, the addition of 20% PVC increased the flexural strength which may be due higher cross linking of PVC particles in HC-PMMA matrix.[21]

Artificial saliva contains 99% water and the water molecules are absorbed by HC-PMMA which reduce the strength of polymerized mass.[22] Findings of the current study are supported by the previous study,[23] which reported that HC-PMMA absorb water when placed in aqueous medium and the water molecules act as a plasticizer in HC-PMMA reducing the flexural strength, hardness and impact strength.

Flexural strength decreased after immersion in water with increasing percentage of PVC except with 20% PVC. It might be due to plasticizing effect of water on polymerized mass of HC-PMMA and PVC blend which is in agreement with previous studies.[22,23] In another study in which the mechanical properties of HC-PMMA were evaluated after aging with different time interval, showed decrease in flexural strength within first week of aging. Immersion of polymerized mass in water causes the leaching of plasticizers, initiators and water-soluble unreacted monomers and micro voids are through diffusion of water molecules.[24]

The amount of dissolved salts in artificial saliva may have an effect on flexural strength of HC-PMMA denture base as reported by Hargreaves, that strong solution of sodium chloride can affect the mechanical properties of acrylic resin.[25] It is possible that the polymerized mass may came in contact with inorganic salts of artificial saliva and caused this some increase in flexural strength of HC-PMMA denture base, as reported in previous study.[26]

Microhardness was significantly increased with increasing percentage of PVC from 5-15% after immersion in artificial saliva. Impregnation of various percentages of PVC in HC-PMMA may have increased the stability of CH bond and methyl groups and hence the rigid polymers can be obtained at higher temperature.[27] Thus the increase in microhardness might be due to the crystallinity of PVC, effect of crosslinking, and structural modification in chain structure of PVC and HC-PMMA.[27]

It was reported by Harrison & Huggett, that microhardness depends on quantity of residual monomer content, that higher residual monomer can adversely affect the microhardness of HC-PMMA denture base resin,[28] but in contrary the terminal boil in short curing cycle might have converted the residual monomers and resulted in increase in microhardness which is supported by the previous studies,[2,6] which reports that the cross linking has an important role in depletion of residual monomer in HC-PMMA.[29]

Immersion in water exhibited an insignificant decrease in microhardness with increasing percentage of PVC in HC-PMMA. Water saturation of denture base resin is although unknown but it might be a two phase process in which soluble constituents (initiators, plastisizers and unreacted monomers) leach out after immersion in water and form microvoids which are filled with water molecules by inward diffusion.[30] Both process – outward leakage and inward diffusion of water molecules are time dependent process which eventually decrease the strength of denture base polymers. reported that microhardness decrease in aqueous environment due to plasticization of water molecules in HC-PMMA denture base supporting the findings of the present study.[31] One of the limitations of this study was that it was in vitro study, since the in vivo conditions are always difficult to be duplicated in the laboratory conditions. Factors involved in the fabrication of specimens such as homogeneity, internal porosities, voids and polishing of the specimens could not be controlled. The cyclic loading of the material was not conducted to simulate the forces produced during mastication.

CONCLUSION

The flexural strength of HC-PMMA denture base resin impregnated with 20% PVC was increased after immersion in water while it had no effect on its microhardness. On the other hand, the microhardness of HC-PMMA denture base resin impregnated with 10% PVC decreased after immersion in artificial saliva..

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AUTHOR'S CONTRIBUTION

Following authors have made substantial contributions to the manuscript as under:

SI: Concept and study design, acquisition, analysis and interpretation of data, drafting the manuscript, approval of the final version to be published.

AH: Analysis and interpretation of data, drafting the manuscript, critical review, approval of the final version to be published.

Authors agree to be accountable for all aspects of the work in ensuring that questions related to the accuracy or integrity of any part of the work are appropriately investigated and resolved.

CONFLICT OF INTEREST
Authors declared no conflict of interest

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DATA SHARING STATEMENT
The data that support the findings of this study are available from the corresponding author upon reasonable request