International Journal of Current Advanced Research
ISSN: O: 2319-6475, ISSN: P: 2319-6505, Impact Factor: 6.614
Available Online at www.journalijcar.org
Volume 8; Issue 10(C); October 2019; Page No.20214-20219
DOI: http://dx.doi.org/10.24327/ijcar.2019.20219.3941

Research Article

EFFECT OF REINFORCEMENT OF DIFFERENT CONCENTRATIONS OF ALUMINA NANO PARTICLES ON DIFFERENT PROPERTIES OF PMMA HEAT CURED ACRYLIC RESINS - AN IN VITRO STUDY

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A R T I C L E I N F O

Article History:
Received 10th July, 2019
Received in revised form 2nd August, 2019
Accepted 26th September, 2019
Published online 28th October, 2019

Key words:
pmma, nanoalumina, hardness, residual monomer.

A B S T R A C T

Aims &objectives: To evaluate and compare the surface hardness (Vickers hardness) and residual monomer content leached out from nano Alumina reinforced heat cure acrylic specimens (i.e, 0%, 2%, 3%, 5% and 7%by weight).
Materials & methods: A total number of 100 heat cure PMMA acrylic resin samples reinforced with different concentrations of nano alumina particles (0%, 2%, 3%, 5% and 7% by weight) were prepared. Among them 50 samples were intended for Vickers surface hardness evaluation by digital microhardness testing machine (METSUZAWA)and they were categorized as Group A, B, C, D & E and the remaining 50 samples were intended for residual monomer analysis by UV-double beam spectrophotometer (SYSTRONICS-2201)and categorized as Group F, G, H, I, & J while Group A and Group F containing 0% wt. nano alumina were considered as controls
Results: The results of the present in vitro study were tabulated and subjected to statistical analysis using one- way, NOVA to know the significant difference between these parameters and Post Hoc test for multiple comparisons of mean differences.
Conclusion: Better surface hardness values are more for higher concentrations compared to lower concentrations of nano alumina particles where as highest mean value of residual monomer content (0.1%) was found in control group-F and least mean value (0.06%) was found in Group-H (i.e., contains 3%wt.nano alumina). The acrylic specimens reinforced with 3% wt of Nano Alumina particles has showed less residual monomer content and moderate surface hardness values compared with other groups

INTRODUCTION

Introduction of nanotechnology into dentistry revolutionized the traditional restorative spectrum of heat cure PMMA acrylic denture base material. The incorporation of these inorganic nano particles into heat cure acrylic PMMA improved its mechanical & physical properties such as its poor strength, particularly fatigue failure inside the mouth, impact failure outside the mouth, lack of radio opacity, poor surface hardness and leaching of residual monomer into oral cavity [1, 2]. In general, properties of polymer nano composites depend on the type of incorporated nano particles, their size& shape as well as the concentration and interaction with polymers, as a result, addition of metal fillers, glass fibres, nano particles etc to heat cured PMMA causes variation of their residual monomer content leading to the change in their mechanical properties where as improvement in its surface hardness prevents the formation of surface irregularities, minimizes micro crack formation & crack propagation leading to lesser denture fractures[3,4]. In the past, many researchers used different weight percentages of nano alumina particles to reinforce PMMA acrylic resins to improve their flexural strength and thermal diffusivity properties etc. but there was limited literature available regarding the evaluation of their surface hardness (VHN) and residual monomer content. Keeping all these considerations mentioned above in priority, the present study was designed to quantify the optimum amount of nano Alumina required for reinforcing the heat cure PMMA acrylics in order to obtain the best possible hardness values and residual monomer content.

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MATERIALS AND METHODOLOGY

This in vitro study was designed to evaluate and compare the surface hardness (Vickers hardness) and residual monomer content leached out from nano alumina reinforced heat cure acrylic specimens (i.e, 0%, 2%, 3%, 5% and 7%by weight) stored in distilled water for 48 hours. This study was under taken at the department of Prosthodontics, Narayana Dental College in collaboration with Central research laboratory, Narayana General Hospital, Nellore and MICROLAB, Ambattur, Chennai.

A total number of 100 heat cure PMMA acrylic resin samples reinforced with different concentrations of nano alumina particles (0%, 2%, 3%, 5% and 7% by weight) were prepared (Fig: 1). Accordingly, these were divided into 2 categories of 50 samples each which were again sub divided into groups A, B, C, D & E (Fig: 2) for evaluation of Vickers hardness and the remaining were categorized as groups F, G, H, I, & J (Fig:3) for residual monomer analysis. The groups A & F containing 0% wt. nano alumina were considered as controls.

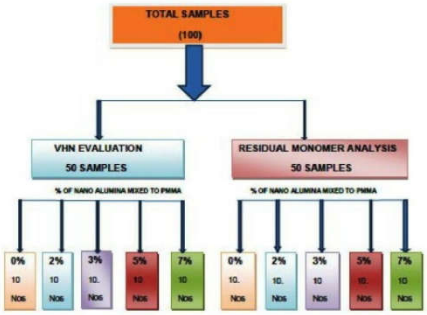


Figure 1 Sample Distribution chat

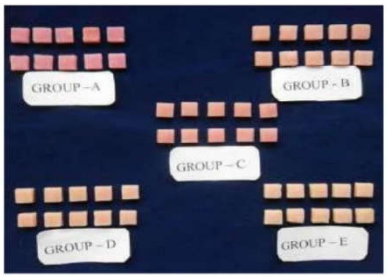


Figure 2 Acrylic Specimens for VHN Testing

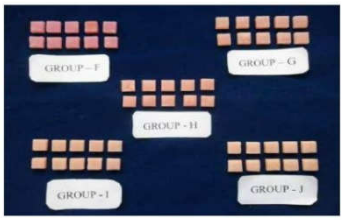


Figure 3 Acrylic Specimens for Residual Monomer Analysis

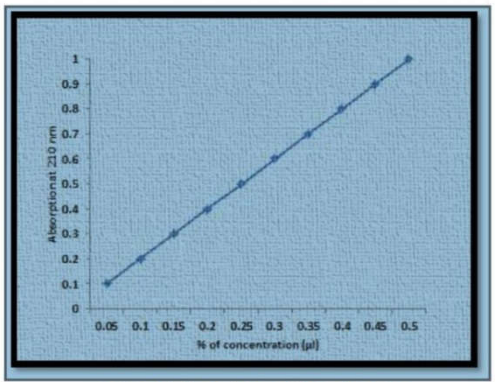
Vickers hardness test was performed using a digital micro hardness testing machine (Model- MMT-X7, METSUZAWA CO.Ltd, JAPAN) by making a total of 3 indentations at different points for each sample (Fig: 4). After withdrawing the indenter, the projected area was viewed under microscope at 400x magnification and area of indentation was calculated by measuring the length of the diagonals of the indentation and the VHN was estimated by using the following formula. By averaging the 3 VHN values, mean VHN of each individual specimen was obtained.

Figure 4 Armentarium for testing VHN

$$VHN = \frac{LOAD}{AREA\ OF\ INDENTATION}$$

Residual monomer content was determined by using a UV-visible light double beam spectrophotometer-SYSTRONICS-2201. After preparation of samples, each specimen was placed in capped glass containers consisting of 10ml of distilled water which is stored at room temperature for 48 hours. Then the solutions were transferred into cuvets and they were subjected to double beam spectroscopy at 210 nm (Fig: 5). The unknown amount of residual monomer leached out into the distilled water was analyzed and values were compared with standard graph prepared by plotting the absorbencies of standard stock solutions of reinforced nano alumina PMMA. (graph1).

Figure 5 Armentarium for measuring Residual Monomer Analysis



Graph 1 Standard Graph

RESULTS

All the measured values of table 1 and table 2 were subjected to statistical analysis by using ANOVA & Post Hoc tests to know the significant difference between all the variables. The mean, standard deviation, mean difference and 'p' values were calculated for all the variables. In the present study $p < 0.05$ was considered as the level of significance. (Table 1 & Table 2)

Table 1 Data of Vickers hardness test of acrylic samples (Hv 25gm)

S.NO	GROUP-A	GROUP-B	GROUP-C	GROUP-D	GROUP-E
1	28.3	28.4	26.8	27.63	27.9
2	28.4	25.6	29.7	27.6	28.1
3	26.6	26.6	27.1	26.6	30.1
4	25.3	28.1	29.2	25.6	29
5	26	29.1	25.4	27.9	26.8
6	25.7	26.6	25.8	27.5	27.6
7	26.6	27.4	28.2	30.9	28.2
8	24.7	24.6	27.6	28.8	31
9	22.9	25.5	29.3	29.7	31.2
10	25.2	26.2	27.2	31	31
AVG	25.97	26.81	27.63	28.29	29.09

Table 2 Data of residual monomer analysis of acrylic samples. (% of concentrations)

S.NO	GROUP-F	GROUP-G	GROUP-H	GROUP-I	GROUP-J
1	0.13	0.07	0.06	0.07	0.10
2	0.13	0.07	0.06	0.07	0.10
3	0.09	0.06	0.03	0.10	0.07
4	0.09	0.06	0.03	0.10	0.07
5	0.10	0.06	0.07	0.10	0.10
6	0.10	0.06	0.07	0.10	0.10
7	0.10	0.10	0.03	0.07	0.10
8	0.10	0.10	0.03	0.07	0.10
9	0.06	0.07	0.10	0.06	0.05
10	0.06	0.07	0.10	0.06	0.05
AVG	0.10	0.07	0.06	0.08	0.08

Stastical analysis of Group- E acrylic samples, (i.e., containing 7 wt% of Nano Alumina) has higher VHN than all the other group of acrylic samples. Similarly residual monomer content gradually decreases from Group F to H and then increases. Highest mean value of residual monomer content is found in Group F and least amount found in Group-H (Table 3 &4)

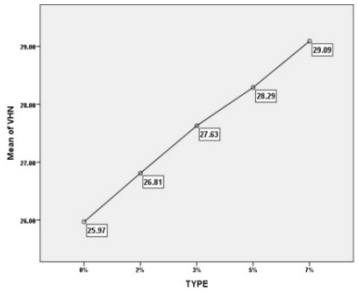
Table 3 Comparison of mean and standard deviation values of Vickers hardness test

S.No	GROUPS	No.of samples	Mean (HV)	Std Deviation	f value	P value
1	A	1O	25.97	1.64		
2	B	1O	26.81	1.43		
3	C	1O	27.63	1.47	5.89	0.001
4	D	1O	28.29	1.79		
5	E	1O	29.09	1.61		

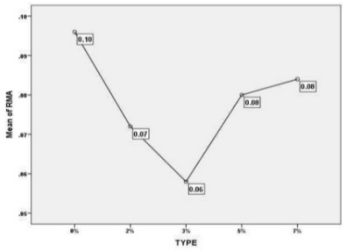
Table 4 Comparison of mean and standard deviation values of residual monomer

S.NO	GROUPS	NO.OF SAMPLES	MEAN (% OF CONCENTRATION)	Std. Deviation	f value	P value
1	F	10	0.10	0.02		
2	G	10	0.07	0.02		
3	H	10	0.06	0.03	4.25	0.005
4	I	10	0.08	0.02		
5	J	10	0.08	0.02		

Graphical presentation of mean VHN values shows the gradual increase in mean VHN value as the weight percentage (i.e. 2%, 3%, 5% and 7%) of nano alumina increased from Group A to Group E. Similarly mean residual monomer values shows decreasing from Group F to Group G & Group H but increasing in Groups I & J, but lesser when compared with control group (Group- F). Highest mean residual monomer was found in Group F (control) and the least value found in Group H. (Graph 2 & 3)



Graph 2 Graphical presentation of mean VHN values



Graph 3 Graphical presentation of mean residual monomer values

Table 5 Comparison of mean difference values of VHN (POST HOC TEST)

Multiple Comparisons			
Tukey HSD			
(X) TYPE	(Y) TYPE	Mean Difference (x-y)	p-value
0%	2%	-.84000	.763
	3%	-1.66000	.154
	5%	-2.32000*	.017
	7%	-3.12000*	.001
2%	0%	.84000	.763
	3%	-.82000	.778
	5%	-1.48000	.247
	7%	-2.28000*	.020
3%	0%	1.66000	.154
	5%	-.66000	.885
	7%	-1.46000	.260
5%	2%	1.48000	.247
	3%	.66000	.885
	7%	-.80000	.793
7%	0%	3.12000*	.001
	2%	2.28000*	.020
	3%	1.46000	.260
	5%	.80000	.793

Table 5 & 6 shows the mean difference values of Vickers hardness and residual monomer of all the acrylic samples. There was a significant mean difference in VHN between A, D and E groups. ($p<0.05$) while significant mean difference in residual monomer values is seen between Groups F and H. ($p< 0.05$)

Table 3 Comparison of mean difference values of residual monomer (POST HOC TEST)

Multiple Comparisons			
Tukey HSD			
(X)TYPE	(Y) TYPE	Mean Difference (x-y)	p-value
0%	2%	.02400	.115
	3%	.03800*	.003
	5%	.01600	.475
	7%	.01200	.730
2%	0%	-.02400	.115
	3%	.01400	.604
	5%	-.00800	.922
	7%	-.01200	.730
3%	0%	-.03800*	.003
	2%	-.01400	.604
	5%	-.02200	.175
	7%	-.02600	.073
5%	0%	-.01600	.475
	2%	.00800	.922
	3%	.02200	.175
	7%	-.00400	.994
7%	0%	-.01200	.730
	2%	.01200	.730
	3%	.02600	.073
	5%	.00400	.994

DISCUSSION

Despite its popularity which satisfy simple processing and ease of repair, the main drawbacks associated with heat cure PMMA acrylic resins as denture base materials are poor strength, particularly under fatigue loads inside the mouth, impact failure outside the mouth, lack of radio opacity, poor surface hardness and leaching of residual monomer into oral cavity [1]. In order to overcome this or to improve its physical and mechanical properties these PMMA resins can be strengthened by reinforcing them with micro sized metal fillers, glass fibres, glass fillers, nano fillers etc[5,6,7,8] . The major difference between nano-metric and micro-metric particles is that nano particles have significantly larger surface area which greatly facilitates the transfer of load from polymer matrix to nano particles, as a result nano particle reinforced hybrid system exhibits higher stiffness and better resistance to wear. Moreover nano fillers showed no reduction in transparency even at the relatively high loadings unlike the micro meter sized filler systems. [9-12]

In the past, many researchers used different weight percentages of nano alumina particles to reinforce PMMA acrylic resins to improve their flexural and thermal diffusivity properties etc [13-16]. But there was limited literature available regarding the evaluation of their surface hardness (VHN) and residual monomer content. Since surface hardness was considered as an indicator of density, Improvement in surface hardness prevents the formation of surface irregularities, which minimizes the micro crack formation, crack propagation leading to lesser denture fractures. Among different tests of surface hardness, Vickers hardness test is considered to be a valid method to evaluate surface hardness of these acrylic resins [17,18]. Similarly the residual monomer (MMA) concentration has been examined widely as it helps in evaluating the biocompatibility of denture base resins, there by evoking hypersensitivity & allergic reactions at higher concentrations (>0.3%) [19,20] and to know the polymerization conversion efficiency which influences several physical and mechanical properties, such as wear resistance and hardness.

The present study was designed to quantify the optimum amount of nano Alumina required for reinforcing the Heat cure PMMA acrylics in order to obtain the best possible mechanical and physical properties like surface hardness and MMA concentration.

In this study results, the reinforcement of PMMA acrylic resin samples with different concentrations of nano Alumina (i.e. 2%, 3%, 5% and 7% by weight) has shown improvement in the surface hardness compared to the control Group (GROUP-A). The highest surface hardness value (Avg. VHN: 29.09) was found in group E, compared to control i.e., group A (Avg. VHN: 25.97) which is higher than average VHN 18 - 25 HV.33, 34, 41. Statistical analysis showed all the mean values were significant and the mean difference values are significant between groups A,D and E. The VHN values gradually increases between the control Group (A) and other Groups i.e. from Group A to Group B (3.2%), Group A to Group C (6.3%), Group A to Group D (8.9%) and Group A to Group E (12%).(Table:5)

The probable reason for this increase in surface hardness might be Al_2O_3 possess strong interatomic bonding, giving rise to its desirable material characteristics and Al_2O_3 is the strongest and stiffest of the oxide ceramics at higher temperatures. Therefore it is expected that when Al2O3 particles disperse in matrix they increase its hardness and strength at higher temperatures.[1] another reason could be the change in powder to liquid ratio because of the inclusion of Alumina Nano fillers. As the filler content increased, the variation in the P/L ratios might affect the mechanical properties of the polymerized resin specimens.[5]

The results observed in this study were contradictory to the investigations conducted by Ihab NS *et al*. (2011),[12] in which author used silanized nano ZrO2 particles to reinforce Heat cure PMMA acrylic resins with different concentrations (i.e, 2%, 3%, 5% and 7% by wt.). In their study surface hardness value increases, but it was statistically not significant. Author explained that when small percentages of Nano ZrO2 particles were added to acrylic resin they were well dispersed in inner parts and only few particles will be involved with the surface of the specimen.

The other parameter evaluated in this study was residual monomer (MMA) leached out from acrylic specimens stored in distilled water for 48 hours after curing. Residual monomer content of acrylic samples gradually decreased in Group-F(0.1%),G (0,07%) to H (0.06%) and then increased up to 0.08% in Group I to J. Highest mean value (0.1 %) was found in Group F (i.e., control Group- contains 0% wt. Nano Alumina) and least mean value (0.06 %) was found in Group H (i.e., contains 3% wt. Nano Alumina), (Table:6). The reason for this changes may be due to addition of low weight fractions (0.5% to 1% wt.) of nano Alumina particles with large surface area, reduces the Glass transition temperature (Tg) of nano composite by 25oC. Further additions of filler (up to 10% wt.) do not lead to additional glass transition temperature (Tg) reductions.[21] As the glass transition temperature reduces, there will be increased mobility of monomer and PMMA particles at high curing temperatures (1000C), so there was more conversion of monomer to polymer leading to a reduction in residual monomer content from group F to H. Thereafter, due to agglomeration of nano particles at higher filler fractions, which interferes with the conversion of monomer to polymer leading to a little increase in residual monomer content in groups I and J.[12,22]. According to the literature available, residual monomer above the 0.23% may leads to allergic reactions.25 But the results obtained in this study has shown residual monomer content less than 0.23% (i.e. below 0.1%) which is within the limitationsThe above results are coinciding with the study conducted by G. Bayraktar *et al*. [7] The author observed that impregnation of glass fibres (silanized) with PMMA / MMA mixture instead of only MMA monomer might prevent the excess residual monomer.

Limitations

Proper homogenization techniques should be followed for even distribution of nano fillers which enhances bonding between the PMMA matrix and nano fillers.

Infrared spectroscopy gives better extreme values of residual monomer content than UV-Visible light double beam spectroscopy.

SEM analysis can reveal the distribution pattern of Nano particles.

The effect of 'AGING' on these materials should be considered.

SUMMARY AND CONCLUSION

Within the limitations of this study, the following conclusions are drawn.

Reinforcement of Heat cure PMMA acrylic specimens with higher concentrations of Nano Alumina particles (Group: E, i.e., contains 7% wt. Nano Alumina) has shown better surface hardness values than the acrylic samples of other groups reinforced with lower concentrations of Nano alumina particles (i.e., 0%, 2%, 3%and 5% by wt.) The mean Vickers hardness values of all the acrylic samples shows 'P' value 0.001, which is highly significant.

Highest mean value of residual monomer content (0.1%) was found in Group-F (control, i.e., contains 0% wt. Nano alumina) and least mean value (0.06%) was found in Group-H (i.e., contains 3%wt.nano alumina).The mean values of residual monomer content of all acrylic samples shows 'P' value 0.005, which is significant.

In clinical conditions where optimal amount of hardness with low residual monomer content was indicated Heat cure PMMA can be reinforced with 3% weight Nano Alumina which has least residual monomer level (0.06%) and better VHN value (27.63).

In the conditions like single dentures, deep palatal vaults, tooth supported, implant supported over dentures etc., which require improved mechanical & physical properties, the Heat cure PMMA reinforced with 7% Wt. Nano Alumina can be advised. But the results of residual monomer analysis showed 0.08% of residual monomer at this wt. percentage of nano Alumina which was greater when compared with other groups, which contain 2% and 3% wt. of nano Alumina. Further, this higher residual monomer can be minimized by other techniques like longer storage in distilled water, immersion in hot water (50 ^{o}C) for one hour before insertion of prosthesis, etc.[23]

A further comparison extended with other concentrations of Nano Alumina for reinforcement of PMMA acrylic resins may give better picture of mechanical & physical properties of these Nano Alumina/ PMMA composites. Further studies should be carried out in clinical situations for better understanding of the nature of these materials and their application in the daily routine practice.

References

1. Mahroo V, Bagheri R, Ali Reza A. Effects of aluminum oxide addition on the flexural strength, surface hardness, and roughness of heat polymerized acrylic resin. *Journal of Dental Sciences*.2012; 7. (3): 238-244.
2. Hamouda IM. Current perspectives of nanoparticles in medical and dental biomaterials. *J Biomed Res*. 2012; 26(3):143-51.
3. Yau WF, Cheng YY, Clark RK, Chow TW. Pressure and temperature changes in heat-cured acrylic resin during processing. Dent Mater. 2002 Dec; 18(8):622-9.
4. Farina AP, Cecchin D, Soares RG, Botelho AL, Takahashi JM, Mazzetto MO, and Mesquita MF. Evaluation of Vickers hardness of different types of acrylic denture base resins with and without glass fibre reinforcement. Gerodontology (2012); 29. (2): e155-e160.
5. Marei MK. Reinforcement of denture base resin with glass fillers. *J Prosthodont*. 1999; 8(1):18-26.
6. Jagger DC, Harrison A, Jandt KD. The reinforcement of dentures. *J Oral Rehabil*. 1999; 26(3):185-94.
7. Bayraktar G, Duran O, Guvener B. Effect of glass fibre reinforcement on residual methyl methacrylate content of denture base polymers. *J Dent*. 2003; 31(4):297-302
8. Sehajpal SB, Sood VK. Effect of metal fillers on some physical properties of acrylic resin. *J Prosthet Dent*. 1989; 61(6):746-51.
9. Hamouda IM. Current perspectives of nanoparticles in medical and dental biomaterials. *J Biomed Res*. 2012; 26(3):143-51.
10. Ellakwa AE, Morsy MA, El-Sheikh AM. Effect of Aluminum Oxide Addition on the Flexural Strength and Thermal Diffusivity of Heat-Polymerized Acrylic Resin. *J Prosthodont*. 2008; 17(6):439-44

11. Ash BJ, Richard WS, Linda S, Schadler. Mechanical behavior of alumina poly/ (methyl methacrylate) nano composites. Macromolecules.2004; 37(4): 1358-1369.
12. Ihab NS, Moudhaffar M. Evaluation the effect of modified nano-fillers addition on some properties of heat cured acrylic denture base material. S. *J bagh coll dentistry*.2011; 23(3):23-29
13. Zhang X, Zhu B, Lin K, Chang J. Mechanical and thermal properties of denture PMMA reinforced with silanized aluminum borate whiskers. Dent Mater J. 2012; 31(6):903-8.
14. Yu W, Wang X, Tang Q, Guo M, Zhao J. Reinforcement of denture base PMMA with ZrO2 nanotubes. *J Mech Behav Biomed Mater*. 2014; 32:192-7.
15. Zhang XY, Zhang XJ, Huang ZL, Zhu BS, Chen RR. Hybrid effects of zirconia nanoparticles with aluminum borate whiskers on mechanical properties of denture base resin PMMA. *Dent Mater J*. 2014; 33(1):141-6.
16. Hasan SB. The influence of alumina nano fillers size and shape on mechanical behaviour of PMMA matrix composite. *J Serb. Chem.Soc*.2014,79(0); 1-19.
17. Anusavice KJ. PHILIP'S Science of dental materials-12th edition, Philadelphia, WB Saunders Co. 2013
18. Farina AP, Cecchin D, Soares RG, Botelho AL, Takahashi JM,Mazzetto MO, and Mesquita MF. Evaluation of Vickers hardness of different types of acrylic denture base resins with and without glass fibre reinforcement. Gerodontology (2012); 29. (2): e155-e160.
19. Yilmaz H, Aydin C, Caglar A, Yaşar A. The effect of glass fiber reinforcement on the residual monomer content of two denture base resins. Quintessence Int. 2003; 34(2):148-53.
20. Tuna EB, Rohlig BG, Sancakli E, Evlioglu G, Gencay K. Influence of acrylic resin polymerization methods on residual monomer release. *J Contemp Dent Pract*. 2013;14(2):259-64.
21. Ash B J, Schadler L S, and Siegel R W. Glass transition behavior of alumina/polymethylmethacrylate nanocomposites. Materials Letters.2002; 55. (1): 83-87.
22. Guo, Zhanhu, *et al*. "Surface functionalized alumina nanoparticle filledpolymeric nanocomposites with enhanced mechanical properties." *J Mater Chem*. 2006; 16.27: 2800-2808
23. Tsuchiya H, Hoshino Y, Tajima K, Takagi N. Leaching and cytotoxicity of formaldehyde and methyl methacrylate from, acrylic resin denture base materials. *J Prosthet Dent*. 1994; 71(6):618-24.

How to cite this article:

MahammadRasool *et al* (2019) 'Effect of Reinforcement of Different Concentrations of Alumina NANO Particles on Different properties of PMMA Heat Cured Acrylic Resins - An In Vitro Study ', *International Journal of Current Advanced Research,* 08(10), pp. 20214-20219. DOI: http://dx.doi.org/10.24327/ijcar.2019.20219.3941

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