

## Effect of ethanol treatment on mechanical properties of heat-polymerized polymethyl methacrylate denture base polymer

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This laboratory study was assessing the nano-mechanical properties (NMP), surface roughness ( $S_a$ ), and topographic changes caused by ethanol on the surface of heat-polymerized denture base polymers at different time past dough stage. Specimens of heat polymerizing acrylic resin (Interacryl Hot, Interdent, Celje, Slovenia) of size 10×10×3 mm were prepared, wet ground, and polished for uniform smoothness and treated with ethanol in concentrations of 40, 70, and 99.9% for 30, 60, and 120 s and statistical analysis was done. Some statistical significance for  $S_a$  were highest with 120 s exposure to 40% ethanol. NMP were the highest for specimens treated with 99.9% ethanol concentration for 120 s, on specimens prepared 30 min past the dough stage. This study suggested that heat-polymerized denture base polymers are prone for changes by ethanol which alters mechanical properties and surface topography. Dough time influenced the ethanol resistance.

**Keywords:** Dough stages, Heat polymerized resin, Nano-mechanical properties, Solvent ethanol, Disinfectant

### INTRODUCTION

Synthetic polymers like polymethyl methacrylate (PMMA) are being used in dentistry, primarily as a denture base material dating back to 1930s<sup>1</sup>. The powder and liquid system based on methyl methacrylate (MMA) and PMMA is mixed at varying ratios to attain a minimum polymerization shrinkage and good handling of the resin mix<sup>2</sup>. Usually the monomer liquid is a mixture of MMA and some dimethacrylates, present in smaller quantities, and acting as a cross linking agent<sup>3</sup>. Biological and mechanical properties of the resin polymer are influenced by the residual monomer that are unreacted or unconverted<sup>4</sup>. Homogenous resin polymer can be obtained by increasing the time of mixing to polymerizing which allows MMA to dissolve the PMMA beads<sup>5</sup>. Between the polymer network and the PMMA beads in an auto polymerized (cold polymerizing) resin an interphase layer is usually seen<sup>6</sup>. The interphase layer which is a so-called semi-interpenetrating network of polymer (semi-IPN) is a cross-linked polymer matrix with a small quantity of dimethacrylate monomer, the major monomer component being monomethacrylate<sup>7</sup>. A higher residual monomer content is seen after polymerization of denture base resins in auto-polymerized resin compared with heat-polymerized resin<sup>8</sup>. On the other hand, the maximum permissible limit of the residual monomer in heat polymerized resins should be less than 2.2%<sup>9</sup>. During the initial days of water storage

of the polymerized denture base it is known to leach residual monomers. Interestingly, cytotoxicity of the resin caused by the residual monomer was reduced when subjected to ethanol treatment without affecting much of the mechanical properties<sup>10</sup>.

Topographic changes in the surface roughness ( $S_a$ ) and the hardness caused by the degradation of the resin, altering the physic-mechanical properties of the polymer are caused to a certain amount by chemical disinfectants<sup>11</sup>. Disinfectants such as ethanol, affect the  $S_a$  of the material to a certain extent<sup>12</sup> and the extent of change in the  $S_a$  serves as an important criteria for bacterial colonization, as  $S_a$  of 0.2  $\mu\text{m}$  and above are critical<sup>13</sup>. Organized cracks or crazing can be seen in thermoplastic resins because of a greater hydrostatic tension and confined yielding, resulting in micro-voids that are caused by some disinfectants<sup>14</sup>. Crazing is linked to the solubility parameter of the chemical and dissolving constant of the polymer, and with ethanol the solubility value is 13 (cal/cm<sup>3</sup>)<sup>1/2</sup> and with acrylic resins 8.9–12.7 (cal/cm<sup>3</sup>)<sup>1/2</sup>. This said, ethanol could be feasible and potential for causing crazing<sup>7</sup>.

The polymer and the monomer when mixed go through a series of stage and will be in the dough stage wherein the monomer diffuses into the polymer. In this case the undissolved polymer can be suspended in a plastic matrix of monomer and at this stage the mix is molded before it reaches the rubbery stage. The hypothesis for this study was that crazing occurs in thermoplastic resins resulting in micro-voids or cracks caused by disinfectants attributed to their solubility parameter, and with ethanol and acrylic

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resins having close solubility parameters prompted us to evaluate ethanol potential for causing crazing. The aim of this laboratory study was to compare and contrast the outcome of solvent like ethanol on the nano-mechanical properties (NMP) and  $S_a$  of heat polymerized acrylic resin (HC) which was molded and polymerized at different post dough stage, *i.e.*, immediate, 15 and 30 min past the dough stage at varying concentrations of ethanol and to evaluate the topographical effect of solvent like ethanol resulting in formation of cracks or crazing. The outcome of the study would be valuable in knowing the polymerizing procedure for better mechanical properties and solvent effect of disinfectants on the surface topography.

## MATERIALS AND METHODS

### Study design

Heat polymerized acrylic resin (Interacryl Hot, Interdent, Celje, Slovenia) was used in this study where the specimens were prepared at 0, 15, and 30 min past dough stage. The specimens were then treated with ethanol (Ethanol absolute, analytical grade, ACS, Reag. PhEur, 2814 batch 15762402) at three different concentrations (40, 70, and 99.9%) and at three different time intervals (30, 60 and 120 s) and were analyzed for  $S_a$ , NMP (nano-hardness and Young's modulus of elasticity), and surface changes (SEM imaging).

### Specimen preparation

The specimens were prepared at room temperature of 23°C, at an atmospheric pressure of 760 mmHg and a relative humidity of 30%. Heat polymerizing acrylic resin specimens of size 10×10×3 mm were prepared and the surfaces were wet ground and polished to achieve uniform smoothness (using a rag wheel and pumice). The monomer (liquid) to polymer (bead powder) was at a ratio of 1:3. Heat polymerized resin was polymerized for 90 min at 74°C, followed by further 30 min boiling at 100°C as recommended by the manufacturer.

Packing of the acrylic denture base material was

done at 3 different times: immediately when the resin reached the dough stage (Group HC1), 15 min past dough stage (Group HC2), and 30 min past dough stage (Group HC3). A total of 200 specimens were prepared for each post dough time and randomly divided into two groups 'S' and 'HC' and named as S1 (0 min), S2 (15 min), S3 (30 min) and HC1 (0 min), HC2 (15 min), HC3 (30 min). Each 'HC1, HC2 and HC3' were subdivided into sub-groups with  $n=15$  and 'S1, S2 and S3' subdivided into sub-groups with  $n=5$  and treated with different concentrations of ethanol for time intervals as shown in Table 1. After treatment the specimens were rinsed with water and dried following which  $S_a$  and NMP were evaluated in 'HC' group and 'S' groups after each treatment were rinsed with water and dried followed by gold sputtering and SEM images were taken.

### Experimental method

In each HC group specimens were evaluated for  $S_a$  and NMP before treatment with ethanol which served as a control group.  $S_a$  before and after treatment of the resin surface at a different concentration of ethanol 40, 70, and 99.9% for time period of 30, 60, and 120 s for specimens prepared at 0, 15 and 30 min after the dough stage was evaluated using a non-contact profilometer (Contour GT, Bruker, Tucson, AZ, USA). Five readings were recorded on both the surfaces of the acrylic resin after each exposure of the specimen for ethanol and time.

Following the analysis of  $S_a$  the specimens were evaluated and measured for the NMP (Young's modulus of elasticity and surface nano-hardness) by a nanoindenter armed with a Berkovich diamond indenter (Bruker). Tests were done at a controlled temperature of 23°C and at low noise conditions. The loading and unloading rate of 0.5 mN/s was used and 10 s of resting period at maximum load, and varying the maximum load between 1 and 50 mN. Five readings were recorded before and after treatment of each specimen surface with ethanol. The Young's modulus of elasticity and nano-hardness are shown in Fig. 1.

SEM images (JSM-6360LV, JEOL, Tokyo, Japan)

Table 1 Grouping of specimens for treatment with ethanol concentration at different time intervals

Ethanol Time Resin	40%				70%			99.9%		
	Control	30 s	60 s	120 s	30 s	60 s	120 s	30 s	60 s	120 s
HC1 (0 min)	HC1A	HC1B	HC1C	HC1D	HC1E	HC1F	HC1G	HC1H	HC1I	HC1J
S1 (0 min)	S1A	S1B	S1C	S1D	S1E	S1F	S1G	S1H	S1I	S1J
HC2 (15 min)	HC2A	HC2B	HC2C	HC2D	HC2E	HC2F	HC2G	HC2H	HC2I	HC2J
S2 (15 min)	S2A	S2B	S2C	S2D	S2E	S2F	S2G	S2H	S2I	S2J
HC3 (30 min)	HC3A	HC3B	HC3C	HC3D	HC3E	HC3F	HC3G	HC3H	HC3I	HC3J
S3 (30 min)	S3A	S3B	S3C	S3D	S3E	S3F	S3G	S3H	S3I	S3J

HC1 and S1; heat cure after 0 min past dough stage, HC2 and S2; heat cure after 15 min past dough stage HC3 and S3; heat cure after 30 min past dough stage.

were taken before and after treatment of the acrylic resin surface for each concentration of ethanol, treated at different time intervals, and prepared at different time past dough stage.

*Statistical analysis*

The results of  $S_a$  and NMP (nano-hardness and Young's modulus of elasticity) with ethanol treatment at different time intervals for groups of 0, 15, and 30 min doughing time were statistically analyzed by two-way and three-way ANOVA and Tukey's HSD *post hoc* tests. *p* Value less than 0.05 was considered as significant. A regression analysis was done between the treatment time and nano-hardness for groups of 0, 15, and 30 min doughing time with a dependent variable (x-axis) as the treatment time by ethanol, and an independent variable (y-axis) as the nano-hardness (GPa). All the data analysis was done using SPSS, version- 23 (IBM, Armonk, NY, USA).

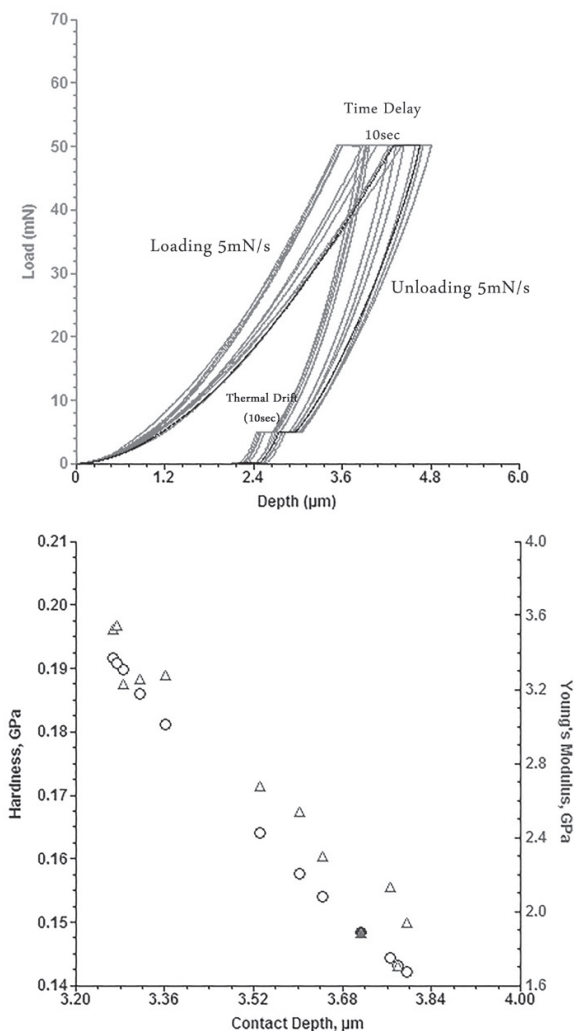


Fig. 1 Graphs illustrating the loading and unloading of the force and plotted values for nano-hardness and Young's modulus of elasticity of heat cured resin of 15 min treated with 40% ethanol.

RESULTS

Ethanol had some effect on the surface roughness, nano-hardness and Young's modulus of elasticity which varied upon the ethanol concentration, dough time and at different treatment time intervals. A reduction in surface roughness was seen in most of the specimen group, 0 min post dough stage specimens treated with 70% ethanol concentration, 15 min post dough stage

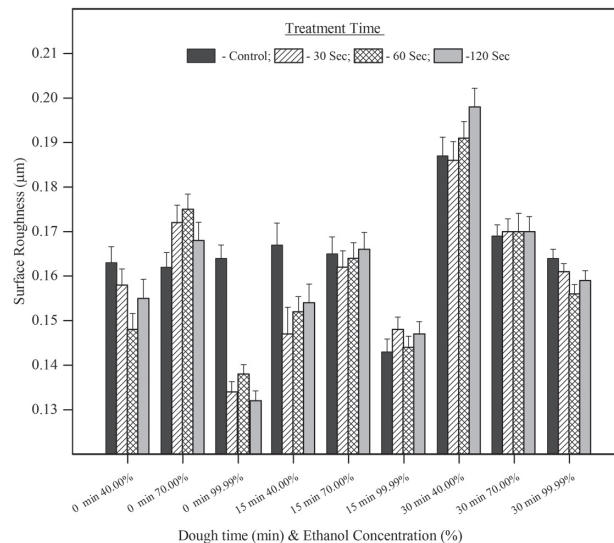


Fig. 2 Graphical representation of surface roughness of specimens prepared at different dough times treated with different ethanol concentration at different time intervals.

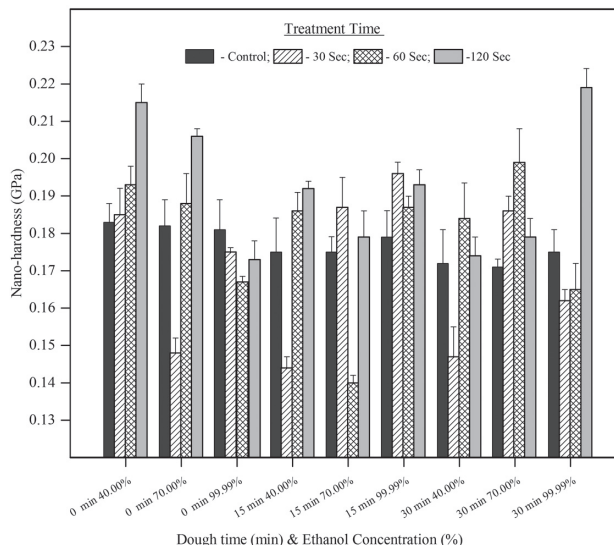


Fig. 3 Graphical representation of nano-hardness of specimens prepared at different dough times treated with different ethanol concentration at different time intervals.

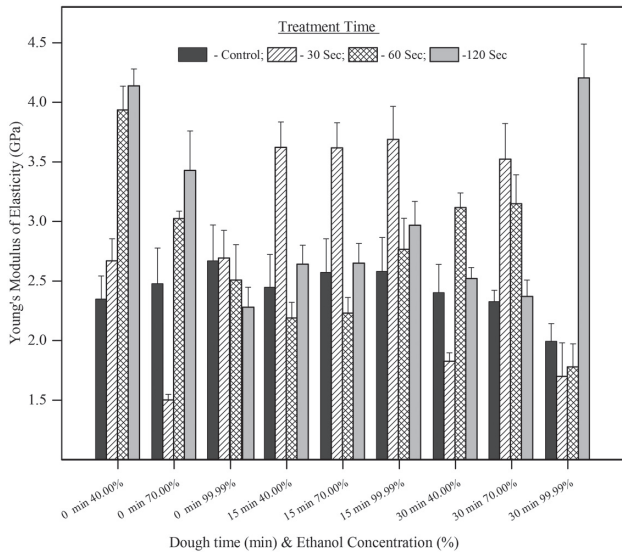


Fig. 4 Graphical representation of Young's Modulus of elasticity of specimens prepared at different dough times treated with different ethanol concentration at different time intervals.

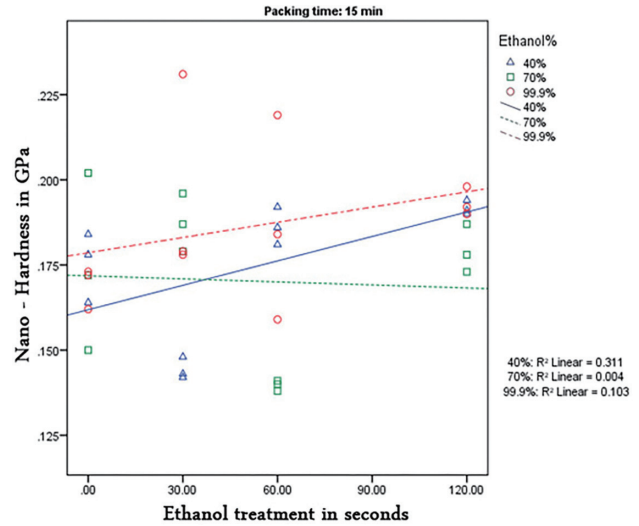


Fig. 6 Regression analysis correlation between ethanol treatment time and nano-hardness for specimens prepared 15 min post dough stage.

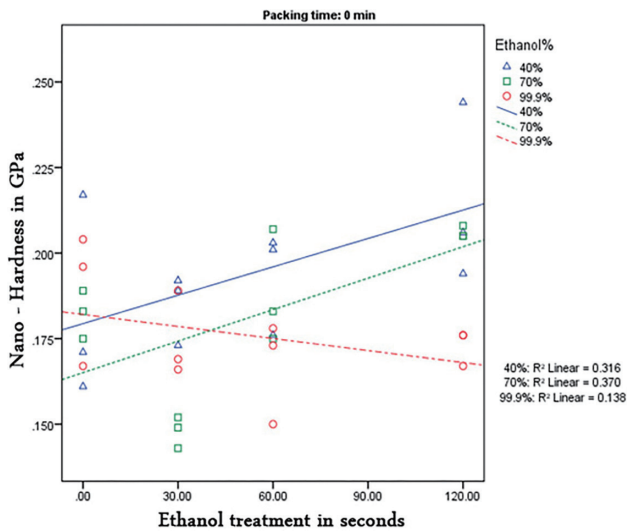


Fig. 5 Regression analysis correlation between ethanol treatment time and nano-hardness for specimens prepared immediately post dough stage (0 min).

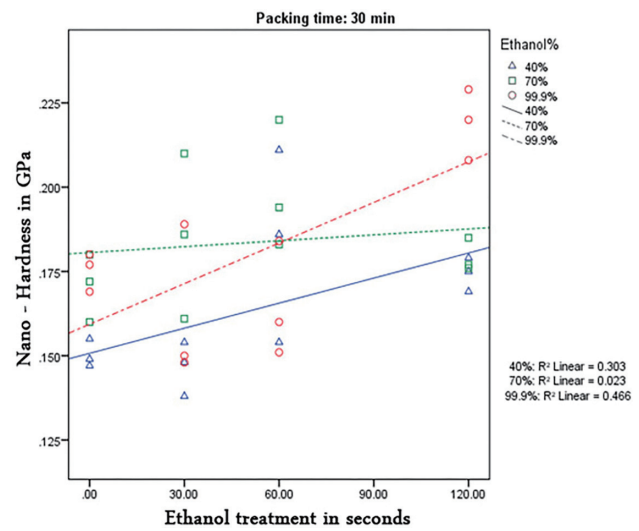


Fig. 7 Regression analysis correlation between ethanol treatment time and nano-hardness for specimens prepared 30 min post dough stage.

specimens treated with 99.99% ethanol concentration, and 30 min post dough stage specimens treated with 40% ethanol concentration showed a slight increase in surface roughness values as shown in Fig. 2. An increase in nano-hardness was seen in all the groups other than those belonging to 0 min post dough stage specimens treated with 99.99% ethanol concentration as shown in Fig. 3. Young's modulus of elasticity was seen to be the least affected for the 0 min post dough stage specimens treated with 99.99% ethanol concentration as

shown in Fig. 4. Regression analysis demonstrated the correlation between ethanol treatment time and nano-hardness for specimens prepared immediately post dough stage for 40% ethanol concentration ( $R^2=0.316$ ,  $p<0.001$ ), 70% ethanol concentration ( $R^2=0.370$ ,  $p<0.001$ ) and 99.9% ethanol concentration ( $R^2=0.138$ ,  $p<0.001$ ) as shown in Fig. 5, specimens prepared 15 min post dough stage for 40% ethanol concentration ( $R^2=0.311$ ,  $p<0.001$ ), 70% ethanol concentration ( $R^2=0.004$ ,  $p<0.001$ ) and 99.9%

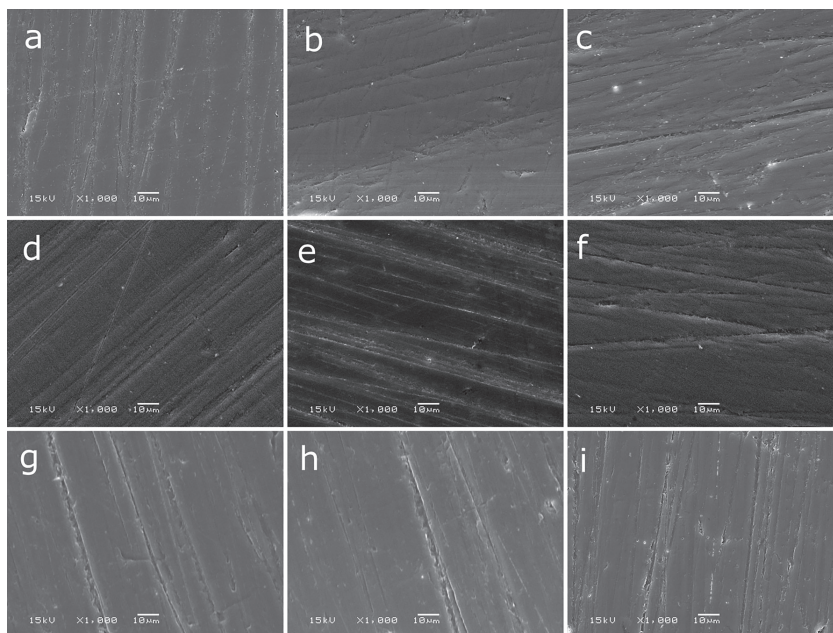


Fig. 8 SEM images of specimens (0 min) treated with 40% (a, b, c; 30, 60, and 120 s, respectively), 70% (d, e, f; 30, 60, and 120 s, respectively) and 99.9% (g, h, I; 30, 60, and 120 s, respectively) ethanol concentration.

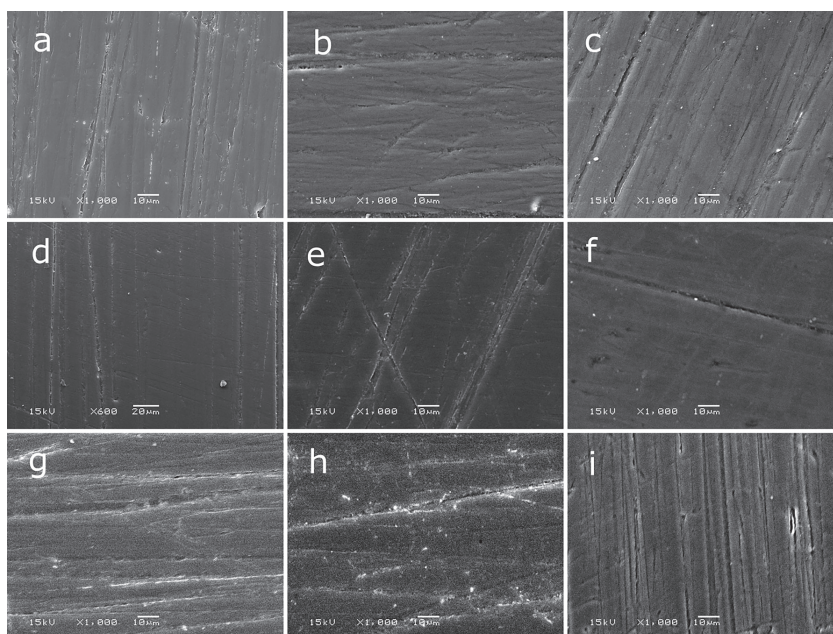


Fig. 9 SEM images of specimens (15 min) treated with 40% (a, b, c; 30, 60, and 120 s, respectively), 70% (d, e, f; 30, 60, and 120 s, respectively) and 99.9% (g, h, I; 30, 60, and 120 s, respectively) ethanol concentration.

ethanol concentration ( $R^2=0.103$ ,  $p<0.001$ ) as shown in Fig. 6, specimens prepared 30 min post dough stage for 40% ethanol concentration ( $R^2=0.303$ ,  $p<0.001$ ), 70% ethanol concentration ( $R^2=0.023$ ,  $p<0.001$ ) and 99.9% ethanol concentration ( $R^2=0.466$ ,  $p<0.001$ ) as shown in

Fig. 7.

SEM images showed slight topographic changes caused by ethanol on the resin prepared at different dough time, treatment time, and concentration groups, as shown in Figs. 8, 9, and 10. Topographical changes

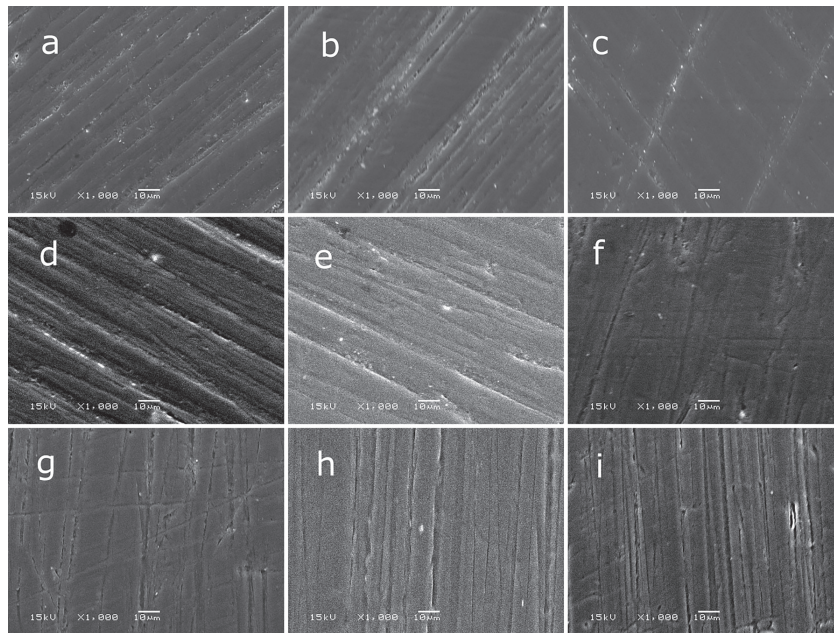


Fig. 10 SEM images of specimens (30 min) treated with 40% (a, b, c; 30, 60, and 120 s, respectively), 70% (d, e, f; 30, 60, and 120 s, respectively) and 99.9% (g, h, I; 30, 60, and 120 s, respectively) ethanol concentration.

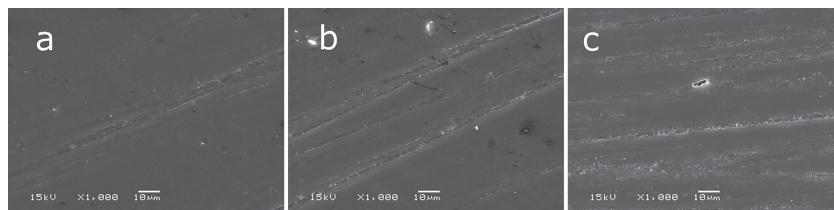


Fig. 11 SEM images of samples without treatment (control).  
a: sample of 0 min of doughing time; b: sample of 15 min of doughing time; c: sample of 30 min of doughing time.

were found in the surface grooves formed during grinding/polishing process. Polymer beads were not visible by SEM images both in control (Fig. 11) and after treatment with ethanol (Figs. 8, 9, and 10).

## DISCUSSION

Denture base polymers which have been polymerized by heat initiation or auto-polymerization vary in their structure, the first one being more homogeneous. More homogeneous polymer structure of heat-polymerized polymer compared to auto-polymerized polymer which is due to the longer period of time when monomer liquid is being in contact with polymer beads, and can thus dissolve the polymer over a longer time<sup>15,16</sup>. In this study, heat cured denture base polymer was selected because there is limited knowledge existing whether the disinfectant ethanol or ethanol used as an intoxicant can have an effect to the surface of acrylic resin dentures.

Changes in the surface structure by ethanol could potentially weaken the denture and enhance microbial and protein adsorption to the denture. If the processing of denture base resin (*e.g.* doughing time) can have an effect, this should be considered by dental laboratory technicians, when dentures are fabricated. Interestingly, in the current study, SEM examination of the polymer specimens did not show any signs of detectable polymer beads even with a short doughing time. However, a light microscope examination has shown the presence of polymer beads even in heat-polymerized denture base polymer<sup>5,17</sup>. In our previous study, we were able to demonstrate clearly visible of PMMA beads and the semi-interpenetrating polymer network (IPN) in auto-polymerized denture base polymer<sup>7</sup>. The dissolving effect of ethanol was most clearly seen to occur in the IPN layer between bead and the matrix polymer. Surface topography changes of this magnitude were not found in the current study with heat-polymerized

denture base polymer. However, some changes were found in terms of NMP, namely in the modulus of elasticity and surface nanohardness.

In this present study the focus was to assess the mechanical properties of heat polymerized acrylic resin at differing concentrations of ethanol on resin molded and polymerized at a different post dough stage, *i.e.*, immediate, 15 and 30 min past the dough stage at different time intervals. The mechanical properties that were evaluated included the NMP (nano-hardness and the Young's modulus of elasticity) and the surface roughness after exposure to ethanol. The important factors governing the post-polymerization effect depends upon the chemistry of the solvent used and monomers<sup>18</sup>, with the former influencing the solubility of the monomers. The correlation between the effect of the solubility of the monomer and the solvent is governed by two important criteria, namely the so-called Hildebrand parameter<sup>19</sup> ( $\delta$ ) and solubility parameter<sup>20</sup>. The  $\delta$  value of the monomer is  $\sim 16.0 \text{ MPa}^{1/2}$ , for ethanol the  $\delta$  value is  $26.0 \text{ MPa}^{1/2}$  and for water  $\delta$  value is  $47.9 \text{ MPa}^{1/2}$ . The solubility parameter of ethanol is  $13 \text{ (cal/cm}^3)^{1/2}$  and for denture base resins is  $8.9\text{--}12.7 \text{ (cal/cm}^3)^{1/2}$ . The  $\delta$  value of ethanol, when compared to that of water, is closer to monomeric methyl methacrylate and this fact may explain the effect of ethanol to be directly proportional to its concentration. The amount of residual monomer basically MMA that was discharged was related linearly to concentration of ethanol<sup>21</sup>. Moreover, the solubility parameters of ethanol and PMMA are closer to each other making ethanol to dissolve polymer linearly and related to the concentration of ethanol. It is noteworthy that denture base polymers contain also minor quantities of cross-linking monomers, such as EGDMA, which is added to the polymer to improve resistance of the polymer against solvent induced crazing<sup>22</sup>. The effect of EGDMA to improve crazing resistance was reported good, but it did not influence water sorption of the polymer.

The polarity of acrylic resins makes them prone to absorb water, depending on the concentration of ethanol. This absorption of water interferes with the polymer chain making the resin to swell and relief of stresses affect the physical properties<sup>23</sup>. Irreversible damage to the resin occurs by scission of the polymer and hydrolytic degradation depending on the time of exposure to imbibed water molecules<sup>24</sup>. The amount of damage to the polymer is governed by the type of polymerizing followed<sup>25</sup> as this influences the monomer to polymer conversion ratio: the, better the conversion, the higher is the amount of elution and less residual monomers<sup>26</sup>.

Dissolution of the solvent into the polymer occurs because of diffusion mechanism and results in salvaging of the polymeric chain. The amount of monomers present in the polymer is directly proportional to diffusion of the solvent resulting in the formation of a swollen gel-like layer, which is seen between the gel layer and the glassy polymer and also between the solvent and the gel layer, plasticizing initially and resulting in crazing

and cracks. The temperature at which the solvent reacts with the polymer has a significant effect in the degree of plasticizing. This said, a high temperature always results in a lesser effect<sup>27</sup>. Quasistationary state is a stage at which no further diffusion of the solvent into the polymer takes place<sup>28</sup>. On the other hand, dissolution of the polymer is influenced by the materials molecular weight and poly or monodisperse within the materials<sup>29</sup>.

PMMA which is indecipherable in ethanol and water alone becomes dissolvable in their mixture<sup>30</sup> forming water cages that have been seen around the hydrophobic ethanol molecules which at 40–50 vol% and at 80 vol% forming hydrogen bond around the ester moieties in the resin polymer<sup>7</sup>. The presence of a cross-linking monomer seemingly changes the dissolution behavior, as does the molecular weight of the PMMA in the beads.

It was found that the solvent/disinfectant ethanol affected the semi-IPN layer between the polymer bead and polymer matrix suggesting; it is more prone for dissolving than the cross-linked polymer matrix or the core of the polymer bead. PMMA polymer beads are syndiotactic polymer and hence resisted dissolving and crazing more effectively compared to that of the semi-IPN structure. In this present study, results for the NMP and surface roughness were consistent and suggested that the use of ethanol in various concentrations could deteriorate denture base polymers topographically. This can be significant at different concentrations and at varying time period. In the near further studies using alternative disinfectants such as sodium hypochlorite, chlorhexidine and/or glutaraldehyde would be important.

## CONCLUSIONS

This laboratory study suggests that heat-polymerized denture base polymers are prone for ethanol induced alteration in the mechanical properties and surface topography. The changes are considerably less in magnitude than as previously been found for auto-polymerized resins. Doughing time influenced the ethanol resistance.

## ACKNOWLEDGMENTS

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## CONFLICTS OF INTEREST

Authors declare that they have no conflicts of interest.

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