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Original Article

# ASSESSMENT OF POLYMERIZATION SHRINKAGE IN RESIN BASED COMPOSITE IMPREGNATED WITH VARIOUS CONCENTRATION OF CELLULOSE NANOCRYSTALS

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## ABSTRACT

*The aim of this research was to determine polymerization shrinkage of resin based composite impregnated with various concentrations of cellulose nanocrystals (CNC). This was an experimental laboratory based study conducted in Peshawar Dental College and Material Research Lab, University of Peshawar from 2020 to 2021. In this study, an experimental composite (EC1) was made and then modified with 0, 2, 4, 6 & 8 wt% CNC. EC1 was taken as the control group, and the interventional groups were designated as EC2, EC3, EC4 & EC5 with 2, 4, 6 & 8 wt% CNC respectively. These were also compared with a commercially available RBC, Coltene (A). For measuring polymerization shrinkage, Archimedes principal was employed. Data was analyzed using One-way ANOVA and Dunnett's test. P value less than 0.05 was considered as significant.*

*EC5 exhibited lowest shrinkage (1.4%) followed by EC4 (1.7%), EC3 (2.3%), EC2 (2.7%) and EC1 (3%) respectively. EC2, EC3, EC4, EC5, showed significantly lower polymerization shrinkage when compared to EC1 (p<0.05). Similarly, polymerization shrinkage for EC4 and EC5 was significantly lower as compared to the Coltene (A) (p<0.05). Based on the results it was concluded that modification of the experimental RBC with CNC significantly decreased its polymerisation shrinkage.*


**Key words:** *Cellulose nanocrystals, Polymerization shrinkage, Resin based composite, Microleakage.*

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## INTRODUCTION

Resin-based composites (RBC) are the leading restorative materials for restoring the damaged tooth structure. The organic matrix components of RBC are formed mainly from methacrylate-type resins like triethylene glycol dimethacrylate (TEGDMA), bis-phenol A-glycidyl methacrylate (Bis-GMA), in which inorganic filler like silica glass, barium and zirconium dioxide particles are present.[1] As, RBC restorations undergo polymerization shrinkage, teeth with such restorations are more susceptible to micro cracking, shrinkage induced stress and eventually leading to post-operative sensitivity or material fracture.[2,3]

Dimensional stability at the junction between tooth and the restoration, plays critical role in the prevention of microleakage. But, most RBCs do not meet this requirement as their dimensional stability is affected by the setting reactions of the matrix.[4] Polymerization shrinkage occurs in all types of RBCs during setting. The stresses at the interface between the RBC and the tooth results in the development of marginal gap that facilitates the entry of fluids and micro-organism. Microleakage can lead to recurrent caries and marginal staining.[5]

Over the years there have been considerable advancements to overcome the short comings of RBCs but polymerization shrinkage still remains a challenge. Several approaches have been adopted to reduce polymerization shrinkage. These include using low-shrinkage dimethacrylate monomers[6,7] or switching to fillers like nanofillers for lower polymerization shrinkage (1.9%).[8] Boulden et.al[9], added thiol-ene-based monomers to resin based composite and reported that increasing thiol-to-ene ratio from 1:1 to 2:1 and 3:1 resulted in

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the reduction of polymerization shrinkage to 2.0 % and 1.8 %, respectively. Son et al[10] reported that by adding barium glass and silica as fillers into resin based composites, further decreased the polymerization shrinkage to 0.78% in 2mm increment as compared to zirconia and silica, which showed shrinkage of 1.46%. Bacchi et al[11] showed that addition of thio-urethane oligomers could reduce development of stress in resin based composites without affecting the general performance. He et al[12], reported that adding 20% phene in RBC reduced the polymerization shrinkage by 1.4%.

The addition of ring opening silorane based molecules has resulted in low shrinkage resin composites. It is synthesized by a reaction between siloxane and oxirane and molecules. The beneficial effect of these composites is due to hydrophobicity of siloxane molecules and ring opening mechanism of polymerization of oxirane molecules. These monomers produce local volumetric expansion because of the opening of ring structure, which compensate for the volumetric shrinkage from C=C polymerization.[26] Another study by Braga and Ferracane has demonstrated that the rate of polymerization and shrinkage induced stress was reduced by increasing the percentage of inhibitor.[27] In another study by Schneider et al., camphorquinone content was replaced partially by phenylpropanedione, which resulted in reduction of polymerization stress.[27]

The use of fibrous fillers was thought to reduce polymerization shrinkage benefiting from the overlapping of the fibers.[30] Increasing filler content also leads to low shrinkage and increased mechanical properties. Composite resin reinforced with short fiber is used as one of dental restoration materials. Incorporating 5% - 7.5% of short fiber fillers into composite resin with filler content of 60% wt. decreases shrinkage by 70%.[28] Oduncu et al [29]reported that the composition of the filler also affect the polymerization shrinkage of composite resin.

Cellulose nanocrystals (CNCs) are important biopolymers with high strength, low density, large surface area, exceptional optical properties and renewability.[13,14] CNCs have gained attention in the field of nanocomposites because of excellent reinforcement and good mechanical properties.[15,16] Wang et al[16] added cellulose nanocrystals/ zinc oxide as functional additives (2, 4, 6, 8 wt %) in dental resin composite to test mechanical and antibacterial aspect of RBCs. They concluded that by adding 2wt % of cellulose nanocrystals/zinc oxide improved compressive and flexural strength of RBC when compared with conventional RBC.

Literature search shows that mechanical properties of the RBCs containing CNC have been tested before, but physical properties such as polymerization shrinkage has not been evaluated. Therefore, the aim of the study is to determine polymerization shrinkage of resin based composite impregnated with various concentrations of cellulose nanocrystals. The null hypothesis of the study was that there would be no difference in the polymerization shrinkage of the resin based composites after the incorporation of CNCs.

## METHODOLOGY

The study was carried out at the Department of Dental Materials, Peshawar Dental College and Material Research Laboratories, University of Peshawar. Description of the experimental composites is presented in Table 2. While different types of materials used for the preparation of dental resin composites are presented in Table 1.

The experimental composite material (EC1) was prepared by mixing Bis-GMA, TEGDMA in a ratio of 3:1, camphorquinone 0.2wt%, 2,6 Di-tert-4-methylphenol 0.1wt%, and N,N-Dimethyl-p-toluidine 0.8wt%. The resin solution was heated in the dark at 60±0.5˚C for 30 mins.[17] For silanization of the silica particles, a mixture of 0.5±0.01 g silane coupling agent, 5.0 ± 0.05g silica particles, 100 ml cyclohexane and 0.1±0.01g n-propylamine was first stirred for 30 minutes at room temperature, and then stirred at atmospheric pressure for additional 30 minutes at a temperature of 60±5ºC. Volatile by-products and solvent were removed by placing the mixture at a temperature of 60ºC degrees centigrade in a rotary evaporator. The obtained silica was then placed in a vacuum oven at 80˚C for 20 hours.[18] The fillers and resin solution thus made were mixed in a ratio of 3:1 wt%.[12] That is 75% of filler with 25% of resin solution. The experimental composite hence made was further modified by replacing silica fillers with 0,2,4,6 & 8wt% of CNC and were designated as EC1, EC2, EC3, EC4 and EC5 respectively (Table 2).

The specimens were prepared based on ISO standard (17304:2013)[19]. A total of ninety specimens were prepared and divided into groups with six specimens each (n=6). Each specimen of mass 1.0 ± 0.10g was moulded into a ball shape, using latex free gloved fingers for all groups of unpolymerized RBCs. For the polymerized groups, the specimens were shaped as previously described, but the mass of each specimen was 0.5 ± 0.10g, (n=12).Twelve specimens of mass 0.5 ± 0.10g were moulded into ball shaped using latex free gloved fingers in each group for polymerized RBCs (n=12).

Polymerization shrinkage in all the materials was determined by Archimedes density comparison principal using the following equation.[12]

Where PS is polymerization shrinkage $D_u$ is the density of the unpolymerized specimens and $D_c$ is the density of the polymerized specimens. Density measurements were determined by using a digital densitometer (Alfa Mirage MD-300 Japan).

The various RBC specimens were polymerised for 40s with light curing unit. The density of the unpolymerised/polymerized specimens was calcualted by using densitometer.

### *Statistical analysis*

Mean and standard deviation values for polymerization shrinkage of specimens in various RBC groups were determined. The data was evaluated statistically using One-way Analysis of Variance (ANOVA) followed by post hoc Dunnet's test using SPSS version 23. P value less than 0.05 was considered statistically significant.

## RESULTS

Table 3 shows the mean and standard deviation values for the polymerization shrinkage of the various RBC groups. One way ANOVA results suggests that polymerization shrinkage of the study groups differ significantly ($p<0.0001$). The findings of this study show that by increasing the concentration of cellulose nanocrystal, polymerization shrinkage decreases. RBC containing 8% CNC (EC5) showed lowest (1.4%) while the RBC group without CNC (EC1) showed the highest (3%) polymerization shrinkage. Pairwise comparison of different groups of composites is presented in Table 4.

In order to check the difference between individual groups, post hoc Dunnett's test was applied which indicated that polymerization shrinkage for EC2, EC3, EC4 and EC5 containing 2, 4, 6 and 8wt% CNC respectively was significantly less as compared to the control group ($p<0.05$), whereas, the polymerization shrinkage for 6 and 8wt% CNC was significantly lower as compared to the Coltene group (A) ($p<0.05$).

## DISCUSSION

The results of this study demonstrated significant differences among the polymerization shrinkage of various composite resin groups leading to rejection of the null hypothesis. Currently, there is a lack of any bench mark for calculating the polymerization shrinkage of resin based composites.[20] Many research papers available have evaluated polymerization shrinkage of resin based composites using different approaches. Since shrinkage percentage rely upon the method used for its calculation, the diversity of the experimental arrangements influences direct comparisons between reported results.[21] Nitta et al. determined the polym-

TABLE 1: MATERIALS USED FOR THE SYNTHESIS OF THE EXPERIMENTAL RBC (EC)

| Materials | Manufacturer |
|---|---|
| Bisphenol A glycerolate dimethacrylate (Bis-GMA) | Sigma-Aldrich USA |
| Triethylene glycol dimethacrylate (TEGDMA) | Sigma-Aldrich USA |
| Camphorquinone (CQ) (photo-initiating agent) | Sigma-Aldrich USA |
| 3 methacryloxypropyltrimethoxysilane (silane coupling agent) | Sigma-Aldrich USA |
| n-propylamine | Sigma-Aldrich USA |
| N,N-Dimethyl-p-toluidine (tertiary amine) | Sigma-Aldrich USA |
| 2,6 Di-tert-4-methylphenol (inhibitior) | Oakwood chemical Columbia USA |
| Silica | Shenzhen Tuoshen technology company China |
| Nano crystalline cellulose | Cellulose Lab Canada |
| Commercially available resin based composite | Coltene universal restoration Switzerland |
| Cyclohexane | VWR chemicals France |

TABLE 2: DESCRIPTION OF THE VARIOUS RBC SPECIMEN GROUPS

| Groups | Description |
|---|---|
| A | Commercially available resin based composite (Coltene universal restoration Switzerland) |
| EC-1 | Experimental resin based composite (75wt% Silica, 0wt% CNC) |
| EC-2 | Experimental resin based composite (73wt% Silica, 2wt% CNC) |
| EC-3 | Experimental resin based composite (71wt% Silica, 4wt% CNC) |
| EC-4 | Experimental resin based composite (69wt% Silica, 6wt% CNC) |
| EC-5 | Experimental resin based composite (67wt% Silica, 8wt% CNC) |

TABLE 3: MEAN PERCENTAGE AND STANDARD DEVIATION VALUES FOR THE POLYMERIZATION SHRINKAGE OF THE VARIOUS RBC GROUPS

| Composite groups | Mean% | ± Std. Dev | Test of Homogeneity of Variances | ANOVA | |
|---|---|---|---|---|---|
| | | | Levene Statistic | F | Sig. |
| Control (EC1) | 3 | 0.25 | 3.229 | 171.5 | <0.0001 |
| Coltene(A) | 2.1 | 0.11 | | | |
| EC2 | 2.7 | 0.08 | | | |
| EC3 | 2.3 | 0.17 | | | |
| EC4 | 1.7 | 0.16 | | | |
| EC5 | 1.4 | 0.17 | | | |

TABLE 4: STATISTICAL ANALYSIS USING POST HOC DUNNETT'S TEST FOR COMPARISONS OF VARIOUS COMPOSITE RESIN GROUPS

| Composites | Mean Difference | 95% Confidence interval | | P value |
|---|---|---|---|---|
| | | Lower bound | Upper bound | |
| Control(EC-1)-Coltene (A) | 0.84 | 0.65 | 1.03 | <0.05 |
| EC1-EC2 | 0.28 | 0.09 | 0.46 | <0.05 |
| EC1-EC3 | 0.62 | 0.41 | 0.83 | <0.05 |
| EC1-EC4 | 1.30 | 1.08 | 1.48 | <0.05 |
| EC1-EC5 | 1.60 | 1.24 | 1.73 | <0.05 |
| A-EC2 | -0.56 | -0.65 | -0.47 | >0.05 |
| A-EC3 | -0.21 | -0.35 | -0.07 | >0.05 |
| A-EC4 | 0.44 | 0.31 | 0.57 | <0.05 |
| Coltene-EC5 | 0.66 | 0.46 | 0.79 | <0.05 |
| EC2-EC3 | 0.34 | 0.22 | 0.48 | >0.05 |
| EC2-EC4 | 1.002 | 0.88 | 1.12 | <0.05 |
| EC2-EC5 | 1.01 | 0.99 | 1.43 | <0.05 |
| EC3-EC4 | 0.65 | 0.49 | 0.81 | <0.05 |
| EC3-EC5 | 0.75 | 0.65 | 1.08 | <0.05 |

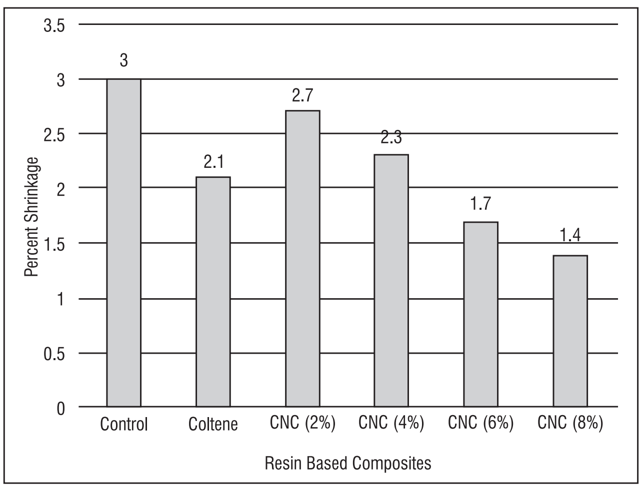


Fig 1: Polymerization shrinkage of various resin based composite groups

erization shrinkage of resin composites using three different approaches; Archimedes' principle, coordinate measuring machine, optical coherence tomography. They reported that the variety of approaches for measuring polymerization shrinkage provided significantly different values for shrinkage.[19]

Despite the incorporation of various new fillers, monomers and initiation systems to improve physical properties of composites, failure of dental composites is very frequent due to bonding disruption.[22] A handsome amount of work is done to address the polymerization shrinkage in RBCs and to improve mechanical properties of various composites based on alteration in the resin phase[23] but the polymerization shrinkage is still frequent. Thus modification in the filler composition and incorporation of CNC of varying strengths was incorporated to address the issue of polymerization shrinkage.

In this study, the polymerization shrinkage of commercially available RBC (Coltene, Switzerland), experimental resin based composite (EC-1, 0wt% CNC) with RBCs groups EC2, EC3, EC4, EC5 containing 2%, 4%, 6% and 8% CNC respectively was compared. After measuring volumetric polymerization shrinkage of each study dental composites, it was found that, polymerization shrinkage for the groups containing 2, 4, 6 and 8% CNC was significantly less as compared to the control group (EC-1), whereas, the polymerization shrinkage for the groups EC4 & EC5 containing 6% and 8% CNC was significantly lower as compared to the Coltene (group-A). EC5 showed significantly less shrinkage when compared to EC4. Lowest polymerization shrinkage was recorded for 8% CNC (mean shrinkage%=1.4) followed by 6%CNC (mean shrinkage%=1.7) followed by 4% CNC (mean shrinkage%=2.3) and 2% CNC (mean shrinkage%=2.7). The results of this study implies that, polymerization shrinkage decreased by increasing the concentration of cellulose nanocrystals in composite resin. The results range between 1-3% which are in accordance with the studies which reported values between 1-3% for high viscosity resin based composite.[24] The results of this study suggests that there might be strong interaction of the CNC with the polymer matrix restricting movement of molecules and that with the increase in the amount of CNC resulted in reduced polymerisation shrinkage, however further investigations are required to investigate the precise role of CNC in polymerisation shrinkage after their reinforcement in resin based composites. Furthermore, it should be noted that with higher concentration of cellulose, mechanical strength of the composites decreases.[16] Hence, based on the results of this work, impregnation of 8% CNC showed maximal reduction in polymerization shrinkage.

The results of our study indicated that the decrease in shrinkage was directly related to the quantity of nano fillers Garoushi[31] and Atai[32] reported that polymerization shrinkage strain reduces by increasing quantity of nanfillers supporting the results of our study. Addition of nanofillers into resin based composite reduced the shrinkable monomers leading to reduced polymerization shrinkage. Incorporating inorganic fillers into the resin-monomers restricts the mobility of monomer-chain resulted in decreased volumetric shrinkage.[33] Another reason for reduced polymerization shrinkage in experimental dental resin composite noted in this study was the type of filler used. A study by Moon et al [34] reported that CNC can improve and control particle-matrix bond strength as well as particle-particle bond strengths respectively, thus limiting the possibility of shrinkage. Lee et al., (2019) in a study observed a 55% decrease in shrinkage in fiber-reinforced cement composites when 0.8vol.% CNC was added, and concluded that for maximum shrinkage reduction and better mechanical strengths, 0.8vol% is the optimum mixing ratio. The findings of our study are also supported by another study[35] which stated that after incorporation of nanocellulose in cement composites, shrinkage can be decreased by up to 18.9% and 5.9%, respectively. It could be further explained by the fact that CNC is an excellent reinforcing agent to the resin matrix only when its amount reaches a optimum value[36]

This work suggests the promising role of CNC in RBC in decreasing polymerization shrinkage associated with composite resin and thus may reduce the incidence of secondary caries. Furthermore, the easy availability of cellulose, its biocompatibility, abundance in nature, transparency and adequate tensile strength make it a favorite choice to be used in dental resin composites[16].

## CONCLUSION

Within the limitations of this study, it can be concluded that modification of the experimental RBC with CNC significantly decreased shrinkage. Based on the findings of this study, resin based composite containing 8% CNC is a suitable percentage which could be used as a filler for reducing polymerization shrinkage

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| | |
|---|---|
| 1 **Fahad Khan Bangash:** | Collected interpreted and analysed the data and prepared the first draft of the manuscript |
| 2 **Amjad Hanif:** | Conceived the idea, supervised and interpreted the data, reviewed and edited the final submitted manuscript. |
| 3 **Mehreen Imran:** | Interpreted the data, reviewed, edited and approved the final manuscript |
| 4 **Hina Rehman:** | Interpreted the data, reviewed and edited the first draft. |