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Influence of nanodiamond incorporation on the impact strength of repaired denture base acrylic resin: an *in vitro* study

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ABSTRACT

Aim. This research aims to evaluate the effects of integrating Nanodiamond on the impact strength of repaired denture base resin.

Methodology. Sixty acrylic resin specimens, sized 50×6×4 mm³ with a v-notch, were created from heat-polymerized acrylic resin. Ten specimens were kept intact while the other 50 specimens were sectioned to half and prepared with 45° beveling and a 2.5mm-repair gap. Nanodiamond particles were added to autopolymerized repair resin and repaired specimens were placed in five categories (n=10) according to Nanodiamond concentrations, unmodified as a control, 0.25%, 0.5%, 0.75%, and 1% ND by weight. Each specimen half was repositioned in a metal jig, and the repair resin was applied to the repair area. Subsequently, all specimens underwent finishing, polishing, and immersion in distilled water before Charpy's impact testing. Data analysis employed Tukey's post hoc test (with significance α=0.05) and one-way ANOVA.

Results. Results showed that in comparison with the control group, the impact strength of repaired denture base resin significantly increased with 0.25% and 0.5% Nanodiamond addition to repair resin (significance at P <0.001). While it insignificantly decreased with 0.75% and 1% Nanodiamond (P=0.108 and P=0.615) and 1% presented the lowest value of impact strength (3.48±0.43 KJ/m^2).

Conclusion. Adding a small amount of Nanodiamond to autopolymerized repair resin demonstrated an increase in the impact strength of repaired acrylic denture base resin. The highest impact strength was recorded with 0.25% ND (6.24±0.55 KJ/m^2), followed by 0.5%ND (5.93±0.21 KJ/m^2), whereas 1%ND exhibited the lowest impact strength value (3.48±0.43 KJ/m^2).

Keywords: analgesic, paracetamol, ibuprofen

INTRODUCTION

Polymethyl methacrylate (PMMA) emerged as a popular choice for denture base material owing to its cited benefits, including convenient fabrication, affordability, aesthetic appeal, easy repair, and comparatively lower toxicity levels [1]. However, PMMA possess inadequate mechanical and physical properties such as low flexural and impact strengths that resulted in denture fracture [2-4]. This eventually affected denture longevity and annoying patients hence more dental visits are required [1]. Surveys have been done on base fracture and reported that 29% of fractured denture associate with midline fracture [5,6] and 68% of denture breaks within a short period after fabrication [7]. Impact failure is a primary cause of this type of fracture due to accidental drop of the denture or exerting more force during denture cleaning [8]. Fractures of maxillary dentures are commonly caused by a combination of fatigue and impact failures, while fractures of mandibular dentures are commonly (80%) caused by impact failure [5,7,9].

Denture repair is preferred over new denture fabrication in terms of being cost-effective and

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time-consuming [2,10]. Reasonable repair should be in the same color as a denture base, dimensionally stable, and obtain the denture's original strength [11]. Successes of denture repair depends on different factors such as repair resin type, repair resin reinforcement, repair surface design, and repair surface treatment [10,12]. In concern repair resin type, autopolymerized repair resin stands out as the prevalent choice in denture repair because of its favorable attributes, such as color matching, easy to manipulate and allowing for chair-side repair [13,14]. Although advantages of autopolymerized repair resin, poor strength was reported and ranged between 18-81 percent of intact heat polymerized denture base [14].

In efforts to enhance repair strength, adjustments in repair surface treatment and surface design have been proposed to bolster repair bond effectiveness [15,16]. Hanna et al. observed that a 45-degree bevel in the repair surface design yielded heightened repair strength [15]. Additionally, treating the repair surface with monomer has been noted to modify the surface structure, thereby increasing the bond at the site of the resin/repair interface [15,16]. The beveling of the surface during repair [15] coupled with monomer application displayed a strong cohesive failure in place of adhesive failure within the repair resin [10,16]. This cohesive fracture pattern within the repair resin indicates its role in the compromised repair strength [10]. Consequently, reinforcement of the repair resin has been recommended using various methods, such as wires, fibers [10], fillers, or nano-fillers [16,17]. Nano-fillers have gained prominence due to their inherent attributes like nano-scale dimensions, significant specific surface area, and effective interaction with organic polymers [18]. The primary objective behind incorporating nanoparticles into dental polymeric materials has been to enhance certain mechanical properties of the resultant nanocomposites [18,19].

Recently, different nanoparticles were suggested in previous studies [16,17,20-22] to improve denture repair strength such as ZrO_2, Al_2O_3, and SiO_2 nanoparticles. Gad et al. discovered that incorporating ZrO_2 nanoparticles resulted in an enhancement of the transverse strength [17]. and impact strength [16]. In efforts to bolster repair strength, the addition of SiO2 nanoparticles was employed, demonstrating that their addition to the repair resin, alongside a 45-degree beveling repair surface, elevated the flexural power of mended resin [20]. Additionally, Tamore et al. observed that augmenting the repair resin with 1% and 1.5% Al_2O_3 nanoparticles notably enhanced the flexural strength in comparison to the unaltered resin [21]. Al-Mahdy and Eltayeb [22] compared the incorporation effect of ZrO_2 and Al_2O_3 nanoparticles into repair resins and found that ZrO_2 nanoparticles increased the impact strengths and flexural of the repaired denture base while Al2O3 nanoparticles decreased.

Nanodiamond (ND) is one of the nano-carbon family and possesses distinctive properties that permit them to be used widely for dental applications [23]. The biocompatibility of ND and its ability to distribute within the acrylic matrix makes it an appropriate reinforcing material for PMMA [24]. ND has been suggested to be incorporated into PMMA heat polymerized denture base resin to improve the properties of final nanocomposite [25,26].

Al-Harbi et al. investigated the impact of varied ND concentrations on PMMA denture base material, finding that higher ND levels decreased impact strength. They suggested employing lower ND concentrations [25]. Similarly, Protopapa et al. noted enhanced mechanical characteristics in temporary restorations using autopolymerized PMMA resin, where they observed reinforced strength with minimal concentrations of ND [27]. Conversely, Fouda et al. highlighted the antifungal properties of PMMA/ND composites, suggesting their potential in managing Candida-related denture stomatitis, a prevalent issue among denture wearers [26].

A positive outcome on the attributes of ND/PMMA composite and on denture repair strength by ND wasn't explored in the previous literature. So, this study was carried out to explore the effect of low concentrations of ND impact the strength of mended denture bases. We formulated a null hypothesis assuming that incorporating ND into auto-polymerized repair resin will not alter the impact strength of the repaired denture base.

MATERIALS AND METHODS

Specimen preparations

60 specimens, measuring 50 mm in length, 6mm in width, and 4 mm in thickness, were created following the standards of ISO (1567: 1999/Amd. 1: 2003(E)) for polymers of denture base [28]. A standard v-notch, 0.8mm deep and spanning the entire 6mm width, was made at the center of each specimen, leaving a thickness of 3.2mm below the notch. Using a customized metal split press mold, wax specimens were prepared accordingly. The acrylic resin specimens were fabricated conventionally for denture base creation. Subsequently, the specimens of wax were placed in a dental flask and subjected to a wax elimination process in a machine for 10 minutes. A separating medium was applied to the stone surface and left to dry. Heat-polymerized acrylic resin (BMS 014 powder) was mixed in accordance to the maker's recommendation and at the dough stage the acrylic resin was packed and pressed under a pneumatic press for 5 minutes and then pressed in the flask clamps for 30 minutes

more. Then, the flask was placed into a thermal curing unit (KaVo Elektrotechnisches Werk GmbH, Leutkirch, Germany) for polymerization using a long polymerization cycle (for the duration of 8 hours at a temperature of 74°C, and after that elevating the temperature for 1 hour at 100°C). After polymerization, the deflasking procedure started after flask reach room temperature and retrieved specimens were finished and polished following conventional method [25]. A digital caliper was employed to assess the specimen 'dimensions. Any specimens found with incorrect dimensions were excluded. The approved specimens' dimensions were then stored in water at room temperature (37ºC) for a duration of 2 days.

Specimens' preparation for repair

Ten specimens remained intact while the other 50 specimens were prepared for repair. A line was drawn at the middle of the specimen to be used as a guide for specimen sectioning into half using a diamond disc. For 45º beveling standardization and 2.5mm repair gap preparation, a silicone jig was used to trim an equal amount from both sides of the specimen. Ultimately, each half had the following specifications: 23.75 mm length of lower surface and an upper surface length of 20 mm with a 45° bevel, a width of 6±0.01 mm, and a thickness of 4±0.01 mm. (Figure 1 A-C). At this stage, two half per specimen were marked for reassembling during repair procedures. (16)

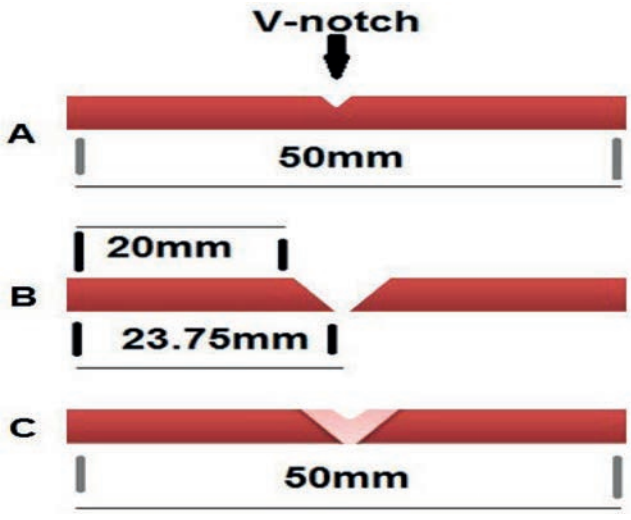

FIGURE 1. Schematic diagram for acrylic resin specimen's dimensions and preparation. A) Intact specimen, B) prepared specimen for repair, C) repaired specimens

PMMA/ND composite preparation

The ND used (Shanghai Richem International Co. Ltd, China) possessed a clarity of 98-99% and size of particles were 30-40 nm, treated as defined in prior research [25,26]. Treated ND particles were measured using a digital scale (S-234, Denver Instrument) to achieve concentrations of 0.25%, 0.5%, 0.75%, and 1% relative to the repair resin powder (Autopolymerized acrylic, BMS 015 powder; BMS Dental). Based on these ND concentrations, specimens were grouped randomly into 5 categories: a control group without addition and 4 test groups containing 0.25%, 0.50%, 0.75%, and 1%ND (Table 1).

TABLE 1. Arrangement of specimens and assigning codes based on Nanodiamond (ND) concentrations

Group/Code	Specifications
I	Intact specimens
Control	Repaired with unmodified repair resin
0.25%ND	Repaired by using the repair resin reinforced having 0.25% Nanodiamond
0.5%ND	Repaired with repair resin reinforced having 0.5% Nanodiamond
0.75%ND	Repaired with repair resin reinforced having 0.75% Nanodiamond
1%ND	Repaired with repair resin reinforced having 1% Nanodiamond

Each mixture was initially mixed with hands and after that stirred with the aid of an electric mixer for a duration of 30 minutes at 400 rpm to attain a uniform dispersal of nanoparticles within the resin powder (reference 25) [25].

Repair procedures

The repaired surfaces underwent a 180-second monomer application, following which the two equal halves of the sample were reassembled in the original molds. Adhering to the manufacturer's guidelines, the repair resin was mixed, slightly overfilling the gap between repairs, and then subjected to pressure at 45ºC. Once fully polymerized, the specimens were taken out of the mold. Any extra resin was eliminated by using traditional denture base polishing and finishing techniques. Subsequently, the dimensions of the specimens were reassessed using a digital caliper, after that it is stored in distilled water for the duration of 72h at 37ºC before testing.

Impact strength test

The impact strength assessment utilized a Charpy's impact testing machine (Digital Charpy Izod impact tester, XJU 5.5, Jinan Hensgrand Instrument Co., Ltd., Jinan, China) (see Figure 2A). The specimen was positioned horizontally onto a metal jig, spaced 40 mm apart within two supports. A pendulum with the weight of 0.5 J was released onto the backside of the specimens, as depicted in Figure 2B. The monitor digitally displayed the energy absorbed upon specimen fracture, enabling the recording of the im-

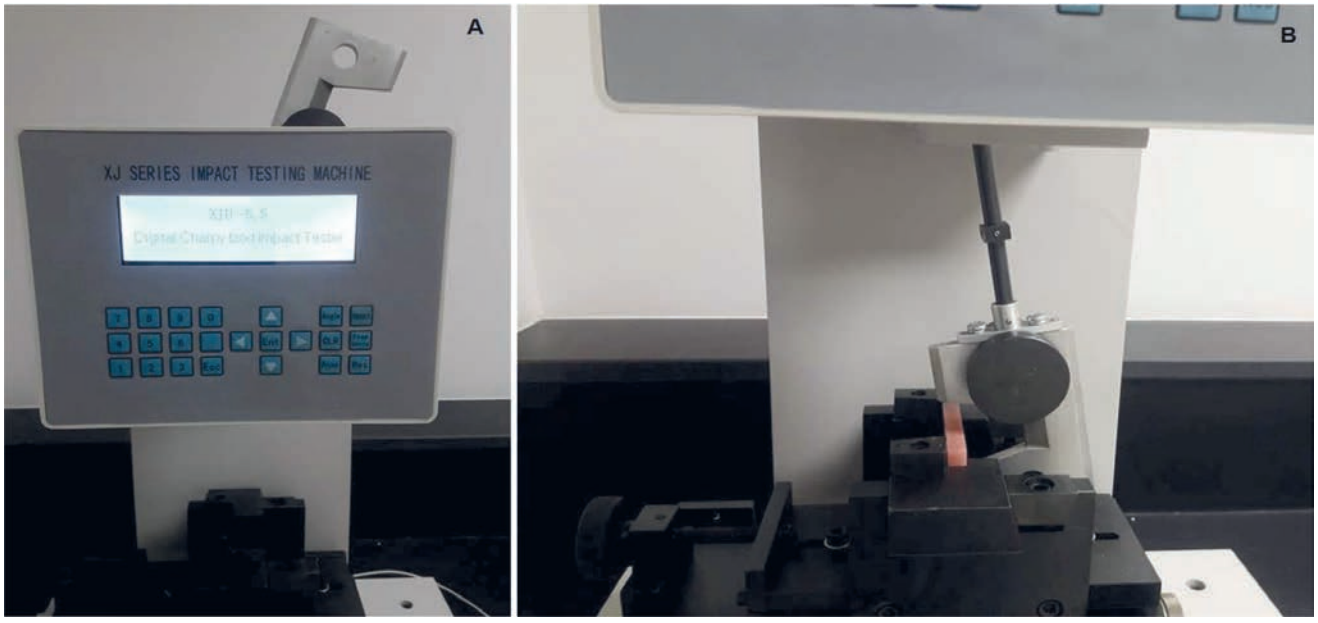

FIGURE 2. Charpy's impact testing machine with loaded specimen

pact strength value (kJ/m^2). The gathered data were organized into tables for future statistical analysis. Post-impact strength testing, the fractured surfaces underwent gold spattering for analysis using Scanning Electron Microscopy (SEM) (FEI; INSPECT S50, Czech Republic) at an accelerating voltage of 20 kV with different magnifications (100x, 500x, 1000, 2000x, and 4000x) as described in previous studies. (16,25)

Statistical analysis

Data was analyzed statistically by using SPSS version 23. Data Normality was tested by using shapiro wilk test and data was found normally distributed. Standard deviations and means were calculated in descriptive statistics. In inferential statistics, one-way ANOVA was used to compare variation in averages between the groups. Tukeys' post Hoc test was used for pair-wise comparison. 0.05 was set as level of significance.

RESULTS

As shown in Table 2, ANOVA analysis showed significant differences between all tested groups (P<0.001). Mean values, standard deviation, and significances regarding Tukey's post hoc test were summarized in Table 3 and Figure 3. In comparison to the intact group, all repaired specimens exhibited a significant reduction in impact strength (P<0.001). A noteworthy decrease was displayed by all repaired groups in terms of impact strength (P<0.001). When compared to the control group, both 0.25%ND and 0.5%ND exhibited a substantial increase in impact strength (P<0.001), while no notable differences were observed between the control and 0.75%ND and 1%ND groups (P=0.108 and P=0.615, respectively). All groups except for 0.25% and 0.5% (P=0.505), presented significant differences. The highest impact strength was recorded with 0.25%ND (6.24±0.55 KJ/m^2), followed by 0.5%ND (5.93±0.21 KJ/m^2), whereas 1%ND exhibited the lowest impact strength value (3.48±0.43 KJ/m^2).

TABLE 2. One-way ANOVA results

Comparison groups	Sum of Squares	DF	Mean Square	F-value	P-value
Intact with ND	88.83	4	22.2	140.17	<0.001*
Control with ND	65.2	4	16.3	103.4	<0.001*
Between ND	53.44	3	17.8	108.3	<0.001*
Intact with all other groups	111.06	5	22.22	144.45	<0.001*

* Significance level at P≤0.05; ND: Nanodiamond

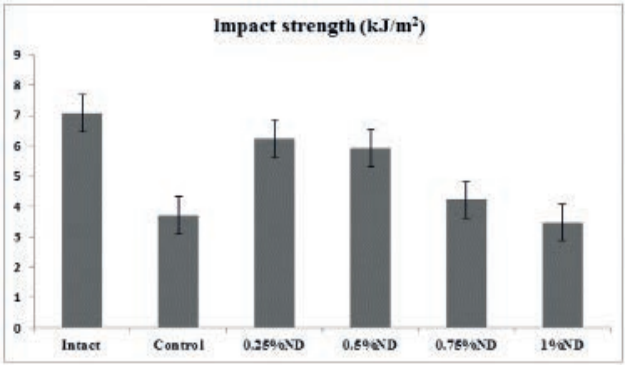

FIGURE 3. Mean value of impact strength for all tested groups

Representative SEM images of fractured surfaces showed different topography with ND addition (Figure 4 A-E). The unmodified specimen (Figure 4 A) a smooth background with small lamellae exhibited

TABLE 3. Mean values, SD, and statistical significances between all groups presented by Tukey's post Hoc test

	Intact	Control	0.25% ND	0.5% ND	0.75% ND	1% ND
Mean ± SD	7.07±0.37	3.70±0.36	6.24±0.55	5.93±0.21	4.22±0.36	3.48±0.43
Intact		<0.001*	<0.001*	<0.001*	<0.001*	<0.001*
Control			<0.001*	<0.001*	0.108	0.615
0.25% ND				0.505	<0.001*	<0.001*
0.5% ND					<0.001*	<0.001*
0.75% ND						<0.001*

* Significance level at P≤0.05; ND: Nanodiamond

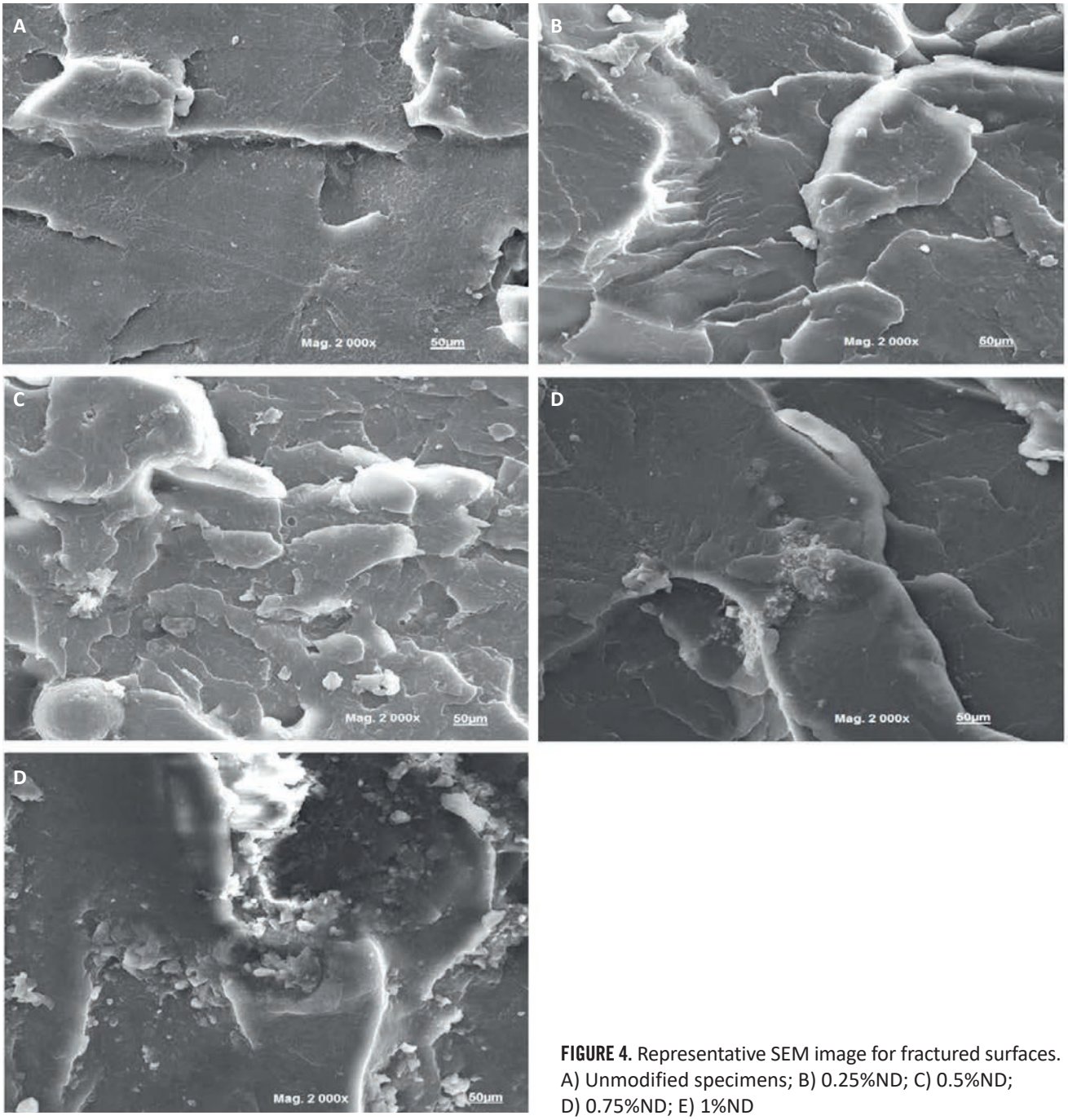

FIGURE 4. Representative SEM image for fractured surfaces. A) Unmodified specimens; B) 0.25%ND; C) 0.5%ND; D) 0.75%ND; E) 1%ND

brittle fracture type. With 0.25%ND and 0.5%ND additions, irregular fractured surfaces with multiple sharp step lamellae (Figure 4 B, C) indicating ductile fracture mode. Moreover, the absence of cluster formations confirmed well distribution of ND particles within resin matrix. With increasing ND concentrations (0.75% and 1%), the fractured surfaces showed less irregular faint lamella and a slightly smooth background with cluster formations of ND particles (Figure 4 D, E).

DISCUSSION

Accidental drop of a complete denture during cleaning, coughing, sneezing, or sudden strokes to the denture is considered one of the major causes of fracture [29]. Therefore, denture base resin is required to have acceptable impact strength to withstand denture fracture and increase its stability [25]. For this study, Charpy's impact test was selected, involving the creation of V-shaped notches on the specimens to simulate frenal notches. These notches were designed to serve as areas of stress concentration. As reported in previous studies, [16,30,31]. The identification of a V-notch validated that the specimens fractured at an identical location during the testing phase. Consequently, it was evident that the surface configuration had an insignificant impact on the strength of the repairs. Based on that, surface design beveling was selected as it allows for increased bonding area in comparison to butt joint [20].

The main causes of denture fracture include accidental drops during coughing, cleaning, sneezing, or sudden impacts. Ensuring the denture base resin possesses adequate impact strength is crucial to withstand these forces and enhance durability [25,29]. This study utilized Charpy's impact test, employing V-shaped notches in the samples to simulate stress concentration areas resembling frenal notches [16]. Previous research confirmed that specimens broke consistently at these notches during testing. [16,30,31], indicating that surface design didn't notably influence repair impact strength. Consequently, beveling the surface was chosen as it provides a larger bonding area compared to a butt joint [20].

While prior research [25,26,32] recommended the use of ND in heat-polymerized PMMA denture base resins, its application in autopolymerized repair resin hadn't been explored. Hence, ND was chosen as a reinforcing material for this study. Consequently, our in-vitro research intended to assess how ND influences the impact strength of repaired denture bases (PMMA). The findings revealed that incorporating various ND concentrations affected the impact strength of the mended denture base resin.

The incorporation of low concentrations of ND (0.25% and 0.5%ND) enhanced the impact strength of the repaired specimens, enhancing their ability to withstand sudden, heavy loads. This aligns with findings by Protopapa et al. [27], who observed a similar impact strength increase in PMMA when ND was added for fixed interim prostheses. The improved strength might stem from the well-distributed ND within the resin matrix, as noted in SEM analysis [25]. Heat treatment of ND generated reactive groups (-COOH, -OH etc.), enhancing interaction between ND and PMMA's carbonyl groups [25,27]. Additionally, the presence of ND particles could alter crack pathways or halt their progression by forming internal cross-linking shear bonds with the polymer matrix [22,33,34].

Al-Harbi et al [25] have been added ND to heat polymerized PMMA denture base materials in concentrations of 0.5%, 1%, and 1.5% and found no significant in impact strength with 0.5% while a significant decrease in impact strength as ND concentrations increased. Consistent with the finding of this study, the impact strength values were decreased with 0.75% and 1% ND insignificantly. This decrease may be attributed to agglomerated ND particle forming loosely attached clusters which may facilitate crack propagation instead of arresting [16,25]. As the clusters of ND increased in number and size, more stress concentration areas led to the breaking of the interactions at the interface and making the de-bonding between resin matrix and ND particles resulted in faster crack propagation [22,34]. This was confirmed by the brittle fracture displayed in SEM analysis. Where faster crack propagation resulted in smooth surfaces without plastic deformation [35]. When this type of crack is started, it will spread instinctively without an rise in the applied stress [25].

Although the selected concentrations of the current study were based on the recommendations of Al-Harbi et al [25] and Protopapa et al, [27] the results of this study confirmed that the ND effect was concentration-dependent and low concentrations showed the highest impact strength values. Therefore, the addition of ND is recommended to be added in low concentrations less than 0.5%.

Looking from a clinical perspective, incorporating low ND concentrations into repair resin notably enhanced the impact strength of mended denture resin bases. However, this current study had limitations, including the use of a single brand of denture base resins and specimens that didn't mimic actual denture configurations. Furthermore, as an in-svitro study, it lacked oral conditions like saliva and chewing forces. Thus, further research exploring various repair resin materials with low ND concentrations under conditions mimicking oral environments is necessary.

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