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The Effect of Different Methods of Nano SiO_2 coating on Thermoplastic Partial Denture Clasp Materials and Their Effect on Hydrophobicity

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ABSTRACT

Objectives: This *in-vitro* study aimed to determine the best coating technique of nano SiO_2 liquid material and its effect on hydrophobicity of thermoplastic partial denture clasp resins.

Materials and methods: 80 thermoplastic disc specimens (Acetal, n=40 and Polyamide, n=40) were fabricated. The specimens were randomly divided into 4 subgroups (n=10/group); brush coating, spray coating, dip coating and uncoated as control group. SEM was used to evaluate the surface topography and the spread of coating material. The subjective method, image quality assessment was used to quantify the images' quality. The mean contact angle was identified by using Image J software.

Results: Mean of opinion score of SEM showed that dip-coated specimens of the two tested materials showed the best coating spread quality among the other coating techniques. Image J software measurements revealed that the contact angle of dip-coated acetal and polyamide specimens exhibited the highest contact angle sequentially in comparison to all tested specimens.

Conclusion: These results suggest that the dip coating technique has the best results in comparison with the two other coating techniques. Nano Sio_2 coating of thermoplastic clasp materials has improved their contact angle which in turn has improved the hydrophobicity of these clasp materials because their external properties are better than those of control specimens. This study will provide a novel external approach for ameliorating thermoplastic partial denture clasp materials using nano

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SiO_2 to enhance their surface properties and solve their limitations and therefore promote the use of these aesthetic clasps.

INTRODUCTION

Retention is one of the critical criteria for the success and longevity of removable partial dentures. Besides effectively serving for retention (Keys., 2017), the extra coronal direct retainer has earned a superior reputation due to being economically and technically easy to manufacture (Fayyaz & Ghani, 2008).

Various metal alloys and polymeric materials have been used for the fabrication of removable partial denture clasps. Nevertheless, there is no material that successfully achieve ideal clasps' requirement. Fulfilling the aesthetic demands is very crucial for the success and prognosis of the removable partial denture especially if the clasps are to be placed facially. For this purpose, thermoplastic resins can be an option as an alternative tooth-coloured partial denture clasp material (Takabayashi, 2010: Polyzois *et al*, 2013). Their advantages of being tooth-coloured, flexible and lightweight would bring pleasant feature and comfort to the patient (Wöstmann *et al*, 2005). Nevertheless, the properties of these materials to be used as denture clasps, clinically are still unknown (Nagakura et al, 2017).

Previously, veneering extra coronal clasps using acrylic resin, composite or silica was among the efforts to camouflage the appalling image of metal (Hansson, 1985: Özcan, 2002). However, discrepancy in choosing the right material to mask the metal is often times causing unsatisfactory outcomes.

The idea of coating specific materials to the base material has improved external characteristics without affecting their internal structure (Wang et al, 2008). Coating of removable prostheses with colloidal silica and polysiloxane has improved scratch resistance and hardness of denture base resin (Chantarachindawong et al, 2012). Using a silicon dioxide SiO_2 coating material over denture resin effectively reduced candida albicans adhesion and growth by lowering the surface roughness (Ra) from (1.14 to 0.56) and enhanced its surface properties including hydrophobicity (Tsutsumi et al, 2023).

As of today, coating of nano SiO_2 over the thermoplastic claps resin may potentially be used to improve the surface properties of the clasp's resin, therefore this study aimed to evaluate the quality of the clasps resin's surface coated with nano SiO_2 in different techniques. Besides, the effect of surface coating on the material's wettability was also observed.

MATERIAL AND METHODS

Specimens preparation

Eighty thermoplastic resin disk shaped specimens at a uniform size (φ20 mm, 2.5 mm thick) were fabricated from acetal (n=40) (Yunnan Co.LTD; China) and polyamide (n=40) (Valplast international corp.; USA) thermoplastic resins.

Wax patterns were invested in plaster moulds. After dewaxing, the specimens of each tested material were prepared using a metallic cartridge. The metallic cartridges were packed with a mixture containing grains of each thermoplastic material singly and 50 mass% of E. glass fibres content. The packed metallic cartridges were heated to plasticize the mixture, then injected into the flasked mould. The mixture was processed by injection moulding technique following their manufactures' instructions using an injection moulding machine (Vertex thermoject 22, Vertex-dental B.V. Netherlands). The materials used in this study and the processing parameters as recommended by respective manufacturers are summarized in (Tables 1 and 2).

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Table 1. Thermoplastic clasp resin materials used in this study.

Material	Type & Abbreviation	Brand name	Colour	Lot	Manufacturer
Acetal	Polyoxy-Methylene (POM)	ZOVGOV	Original white	Q/YTH 004-2019	Yunnan Co.Ltd, China
Polyamide	Valplast (Nylon)	Valplast®	Original pink	022284200111	Valplast International Corp., USA

Table 2. Processing parameters employed for the two thermoplastic clasp resin materials.

Material	Heat temperature (°C)	Heating time (min)	Pressure (MPa)	Flask temperature (°C)
Acetal	220	20	4.0	96
Polyamide	288	9.30-12.30	1.0	100

Finishing and polishing

Each specimen was trimmed from nodules and irregularities using tungsten carbide bur, followed by finishing using silicon carbide waterproof abrasive paper at grits of 280 and 600 for 10 seconds each. Dry polishing was done on one surface of each specimen, using soft and superfine brushes respectively for 30 seconds using a handpiece (Kavo Smart Air plus type 4941; Germany) rotating at 10,000 rpm (Soares et al, 2019). While the other surface was left unpolished. A serial number was given for each specimen to prevent multiple testing (Al-Kheraif, 2014).

Nano SiO_2 coating

A very thin layer of silane coupling agent (Bis-silane™; Bisco USA) was applied on the etched tested surface of each specimen. One coat of the silane coupling agent was applied using (Bis-dark blue brush applicator, (Bisco USA) to promote the bonding between thermoplastic resin materials and nano SiO_2 coating material (Matinlinna et al, 2017).

Both acetal and polyamide thermoplastic resin specimens were divided randomly into four subgroups of ten specimens each. The groups for each material were designated in the following order. The first subgroup specimens were coated with nano SiO_2 liquid using a brushing technique, (Bis-dark blue brush applicator, Bisco USA). The brush was applied to coat specimens in one direction for 20 seconds (Sayed et al, 2023). The second subgroup specimens were coated using a dipping technique, the specimens were dipped for 20 seconds in a container containing nano SiO_2 liquid horizontally and they were arranged separate from each other (Maleki et al, 2023). The third subgroup specimens were coated using a spray technique. A professional mini air brush gun (T-130 shenzhen tomtop technology Co.Ltd, Guangdon China) was used to spray the nano SiO_2 liquid perpendicularly and at a 10 cm distance to the specimens in one direction for 20 seconds (Liang et al, 2023). While the fourth subgroup specimens were left uncoated as a control group.

https://doi.org/10.24191/cos.v11i1.26034

In achieving a uniform thickness for all coated specimens, the dry coating thickness was measured using digital coating thickness gauge (HW-300S; Vakind - China). Three measurements were made at three different locations on each specimen and the mean value was calculated (Ng et al, 2021).

Surface topography and coating spread

The surface topography and the efficacy of the coating technique to spread the coating material on all test specimens surfaces were measured. All specimens were stored in distilled water for 48 hours in an incubator (Binder-Germany) at 37°C for rehydration and completion of polymerization (Gad et al, 2022). Specimens were dried using paper tissues before testing (Kumavat et al, 2016).

Scanning electron microscope (SEM) (TM3000; HITACHI Corp. - Japan), was used to assess the surface topography and the amount of coating material spread of all test specimens. Prior to testing, specimens were gold sputtered to make them conductive. Each test specimen was assessed using 500 and 1000 magnification power respectively (Tsutsumi et al, 2023). Four different categories were assigned to express the coating quality of all specimens comprised of (1=very dissatisfied, 2=dissatisfied, 3=satisfied and 4=very satisfied). Every specimen gained its score based on homogenous appearance, absence of irregularities, absence of rough areas and absence of scratches or cracks (Bornes et al, 2023). The assessment was repeated on another day for reliability (Jafari et al, 2017; Silva et al, 2022).

Measurement of wettability (contact angle)

The wettability (contact angle) of specimens was measured after storage in distilled water for 48 hours in an incubator (Binder-Germany) at 37°C for rehydration and completion of polymerization (Gad et al, 2022). Every test specimen was dried using paper tissues before the measurement was carried out (Kumavat et al, 2016).

Stagnant drop method was utilized to measure the contact angle of specimens, using Image J software. A small droplet of distilled water of 10µL was formed on the tested side of the specimen using fine needle (Han et al, 2022). After that, the photograph of droplet on the specimen's surface was taken using a 64 MP digital camera of an android smartphone (Galaxy S20; Samsung – South Korea). The camera of the smart phone was equipped with an external macro lens (APL-0.45WM; Apexel – China). Photographs of samples were taken at a fixed distance of 5cm for all test specimens and at the same horizontal level using adjustable mobile phone holder (400521-A; Onsmo – China) (Han et al, 2022). Then, all measurements were performed under a standard illumination corresponding to average daylight (Committee JIS, 2009). All the images of the contact angle were uploaded to Image J software version 1.46 (NIH., Maryland - USA) for contact angle determination. The measurements were performed in triplicate and the mean of contact angle value was calculated.

Statistical analysis

The data were statistically analysed using SPSS software program version 27 (IBM Corp., Armonk - NY) for Windows. Shapiro-Wilk test showed normal distribution for all the values.

Two-way analysis of variance (ANOVA) was used to inspect the difference in contact angle means between specimens of the four subgroups of each tested material respectively and between specimens of the eight subgroups of the two tested materials. The analysis showed a significant variation in means at ($p<0.0001$), followed by (LSD) post hoc test which revealed a significant difference in angle means between the four tested subgroups of each material at ($p<0.0001$). There was a significant interaction between the effect of coating techniques and the material type (F 7.72= 507.536) and ($p = <0.0001$). Dip coated acetal resin specimens exhibited the highest contact angle mean (105.6095°), while non coated polyamide resin specimens displayed the lowest contact angle mean (87.7468°). There was no significant difference between brush coated specimens and spray coated specimens of the two tested materials respectively.

Chi-square test was used to statistically analyse the variations in coating quality spread between specimens of the four subgroups of each tested material. The analysis showed statistically significant difference in coating quality within the subgroups of the same material and between subgroups of the two tested materials at ($p<0.05$).

RESULT

Quality assessment of SEM image

The Chi-square test results are summarized in Table 3, which showed a significant variation between non coated (category 1), brush coated (category 3), dip coated (category 4) and spray coated (category 3) of the two tested materials using nano SiO_2 coating liquid for thermoplastic resin partial denture clasp materials. Brush coated and spray coated specimens exhibited the same coating quality. The dip-coated specimens of the two tested materials showed the best coating spread quality among the other coating techniques.

Table 3. Coating quality results

Groups	Coating quality, n (%)			X^2	P-value
	Very dissatisfied	**Satisfied**	**Very satisfied**		
Non coated acetal resin	10/(100)	0	0	127.815	<0.0001*
Non coated polyamide resin	10/(100)	0	0		
Brush coated acetal resin	0	10/(100)	0		
Brush coated polyamide resin	0	10/(100)	0		
Dip coated acetal resin	0	0	10/(100)		
Dip coated polyamide resin	0	0	10/(100)		
Spray coated acetal resin	0	10/(100)	0		
Spray coated polyamide resin	0	10/(100)	0		

The surfaces of the dip coated specimens of the two tested materials appeared homogenous, regular, free from cracks and smoother than the surfaces of other coated groups of the two tested materials and control group which seemed heterogeneous, irregular, rough and scratched, (Figure 1 and 2).

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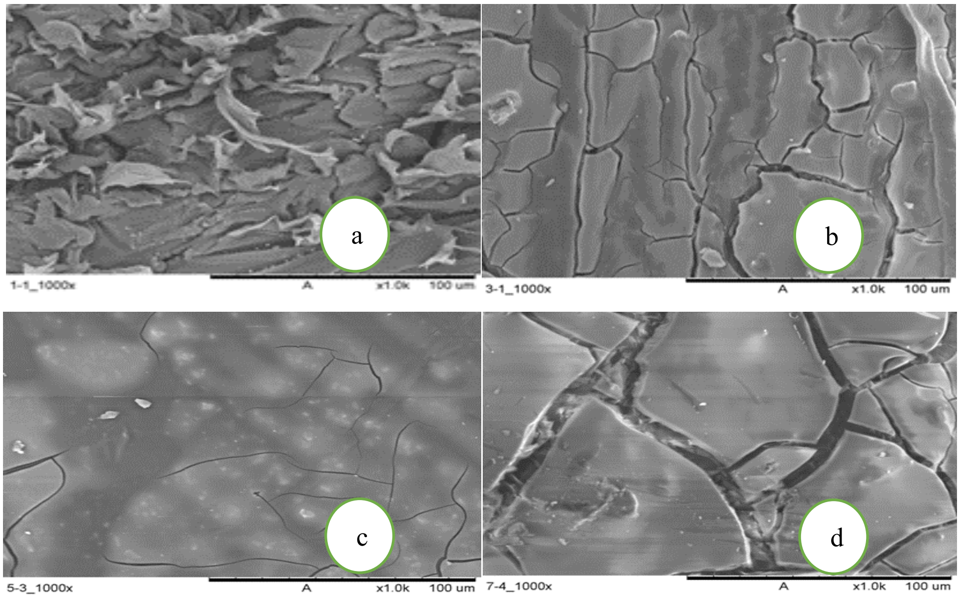

Fig. 1. Acetal resin surface photomicrographs using SEM : (a-Non coated, b-Brush coated, c-Dip coated, d-Spray coated)1000x magnification.

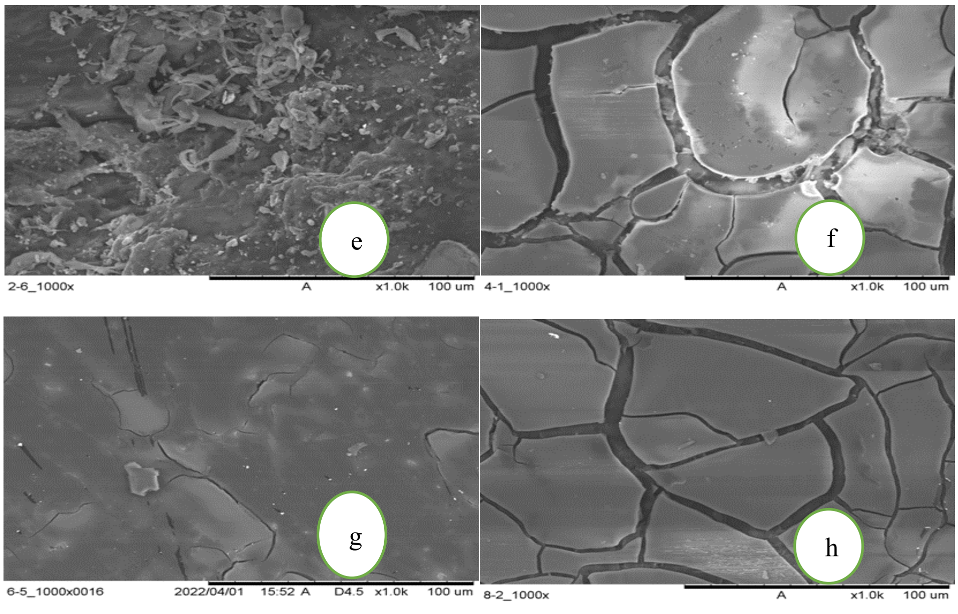

Fig. 2. Polyamide resin surface Photomicrographs using SEM (e-Non coated, f-Brush coated, g-Dip coated, h-Spray coated).

Contact Angle of Acetal and Polyamide clasp resin materials

The two-way ANOVA results are summarized in Table 4 showing a significant difference in contact angle means between the four subgroups of each tested material ($p<0.0001$). The least significant difference (LSD) post hoc test was used to test the pairwise comparison of all means and found all the groups were

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significantly different (p<0.0001). Between the resin group, irrespective of the coating techniques used, acetal resin showed a higher mean contact angle in comparison to the polyamide group. Among the coating techniques, dip-coated acetal resin and polyamide resin exhibited the highest mean contact angle; 105.61 ± 1.02 and 100.79 ± 1.09, respectively in both acetal and polyamide groups. Non coated acetal resin and polyamide resin revealed the lowest mean contact angle; 90.12 ± 0.30 and 87.74 ± 0.91 respectively. Brush coated specimens and spray coated specimens of the two tested materials respectively showed convergent contact angle results; (95.54 ± 0.62 and 96.20 ± 0.95) for acetal resin, and (92.91 ± 0.66 and 92.93 ± 0.57) for polyamide resin.

Table 4. Contact angle mean of acetal and polyamide resins coated with SiO2 using different techniques.

Type of group	Material	Mean	Std. Deviation	N	P value
non coated acetal resin	acetal resin	90.1240	.30167	10	<0.0001
non coated polyamide resin	polyamide resin	87.7468	.91484	10	
brush coated acetal resin	acetal resin	95.5490	.62039	10	
brush coated polyamide resin	polyamide resin	92.9160	.66309	10	
dip coated acetal resin	acetal resin	105.6095	1.02178	10	
dip coated polyamide resin	polyamide resin	100.7912	1.09315	10	
spray coated acetal resin	acetal resin	96.2047	.95393	10	
spray coated polyamide resin	polyamide resin	92.9399	.57552	10	
Total	acetal resin	96.8718	5.68989	40	
	polyamide resin	93.5985	4.78780	40	

* Two-way ANOVA (total n=80, n=10/group) with LSD post hoc analysis: significant difference among all the specimens groups, p<0.000

The surface quality and contact angle means of all tested groups are illustrated in (Figures 3 and 4).

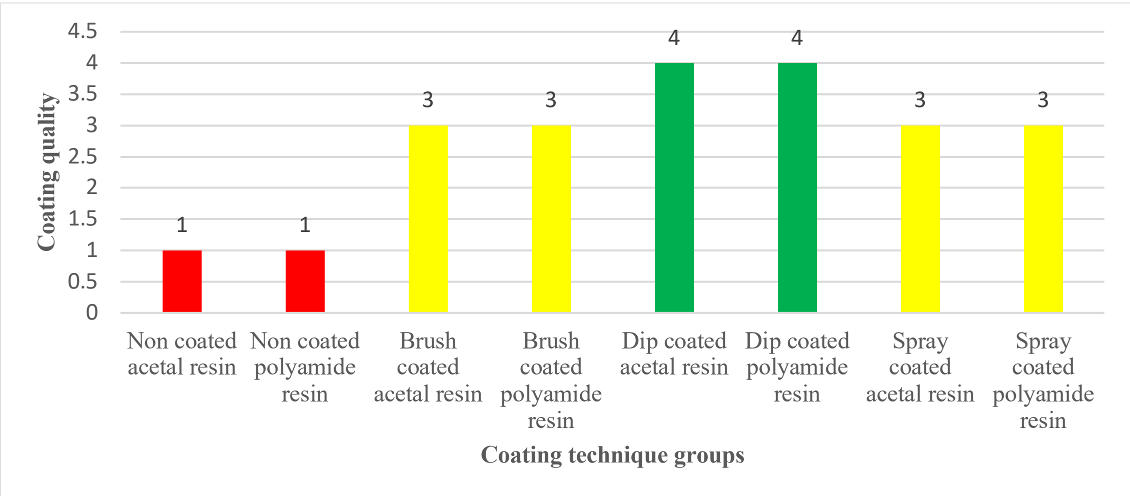

Fig. 3. Surface quality of (non coated, brush coated, dip coated, and spray coated acetal and polyamide groups).

https://doi.org/10.24191/cos.v11i1.26034

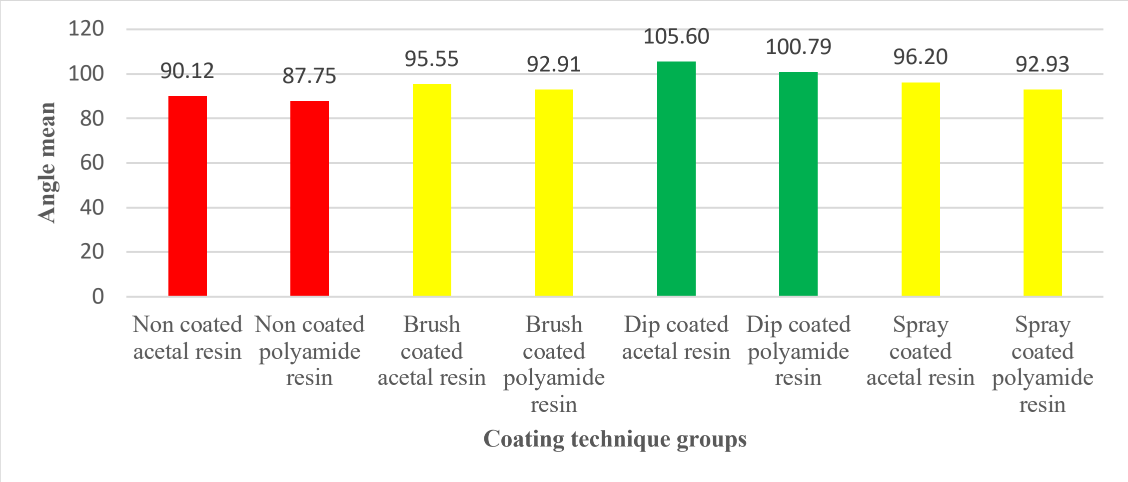

Fig. 4. Mean contact angles of all tested groups.

DISCUSSION

The effect of nano SiO_2 coating technique and its effect on wettability was investigated in this study. SEM was used to assess the surface topography and the efficacy of each coating technique to spread the coating material on all test specimens surfaces. Among all techniques used, dip coated specimens have shown better surface quality with the highest hydrophobicity regardless of the type of resin.

Dip coated specimens of the two tested materials had the best coating spread quality among the other coating techniques which can be attributed to that dip coating technique allowing distinguished dispersion of sufficient coating material amount over wide surface area without any presence of scratches or cracks (Gad et al, 2022). The surfaces of the dip coated specimens of the two tested materials appeared smoother and flatter than the surfaces of other coated groups of the two tested materials and control groups which seemed rough and scratched, this is ascribed as a direct consequence due to the uniform dispersion, good distribution and thoroughly penetration of the embedded SiO_2 nanoparticles which in turn could be considered as an evidence regarding the efficiency of the dip coating technique (Ammar et al, 2016). Additionally, it has been proven that the dip coating technique is ideal for coating small pieces or parts used for protective coatings which are compatible with this experiment specimens shapes and purposes (Caswell, 2014).

Brush and spray coated specimens surfaces of the two tested materials showed some scratches and cracks of the same magnitude and extent which reflects the utilization of improper coating techniques due inferior dispersion of adequate coating material and lack of uniform spread and distribution of the nano coated SiO_2 coating material over coated specimens surfaces. Brush coating technique has some disadvantages which are in agreement with this experiment results such as lack of consistency which resulted in areas of the specimens not being properly coated and presence of scratches and cracks that produces low quality coating (Caswell, 2014). Furthermore, it has been observed that the brush bristles create some marks over the coated specimens surfaces which is both functionally and aesthetically unacceptable. Spray coating technique requires the use of special spray gun which converts the nano SiO_2 coating material liquid in to a mist that is scattered over coated specimens surfaces leading to producing bad dispersion and inconsistent distribution of the coating material over coated specimens surfaces which is in harmony with results of this experiment. It is worth mentioning that the use of spray coating technique is combining complexity (Caswell, 2014), inefficiency and unreliability (Fotovvati et al, 2019).

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In this experiment the use of 2 wt. % of nano SiO_2 indicated two advantages, the first advantage is facilitating the capability of the coating technique whereas the second advantage is this concentration is sufficient to spread the coating material all over the surface area of experiment specimen without the creation of any separation or gaps. This is because, the space between the nanoparticles becomes smaller as the number of the particles increases within the coated area through the prevention of formation of large agglomerated particles of the coating material which was found in strong agreement with SEM surface Photomicrographs of this experiment and results of previous studies (Ammar et al, 2017).

In Contact angle (wettability) measurement experiment the mean contact angle of dip coated acetal resin and polyamide resin specimens exhibited the highest mean contact angle sequentially in comparison to the other coated groups of the two tested materials and control groups.

The dip coated specimens of acetal resin and polyamide resin showed the highest mean contact angle sequentially in comparison to the other coated groups of the two tested materials and control groups which reflects their hydrophobicity. This is due to the effect of dip coating technique to raise surface roughness of coated specimen surfaces through formation of air pockets between the water droplets and the coated specimen surface. It should be noted that, the dip coated specimens of acetal resin gained the highest mean contact angle in comparison with other coated groups of the two tested materials and control groups because they naturally shown to have little hydrophobicity so the dip coated technique together with the nano SiO_2 coated material added additional value to their hydrophobicity, the mean contact angle for non- coated acetal resin was 90.12º then increased to 105.60º after dip coating with nano SiO_2 coating material.

The characteristics of 2 wt. % of nano SiO_2 can be utilized to explain the increased contact angle values of the dip coated specimens of acetal resin and polyamide resin sequentially. This is because the nano SiO_2 coating material possesses a low surface energy which increased the contact angle of the coated specimens surface and therefore increased their hydrophobicity (Ammar et al, 2017).

The limitations of this study include using a single type of coating technique for each tested group, and absence of the conventional laboratory method for contact angle measurement. Therefore, further investigations using a combination of more than single coating technique are recommended to investigate the effect of using combined coating techniques for each tested group and the use of the conventional laboratory method for contact angle measurement for more reliability and sureness.

CONCLUSION

Based on these results and within the limitations of this study, it can be concluded that, dip coating technique has significantly improved the surface properties of thermoplastic resin clasp materials in comparison with the two other coating techniques. Coating thermoplastic resin clasp materials using nano Sio_2 is practically effective in respect to improving the hydrophobicity properties of these materials through increasing the contact angle values of these coated materials surfaces and therefore improving their wettability because their external properties are better than those of non-coated control specimens.

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CONFLICT OF INTEREST DECLARATION

We declare there is no conflict of interest in carrying out the work.

AUTHORS' CONTRIBUTIONS

Ayoub Zabida conceptualised the central research idea, provided the theoretical framework, carried out the research and laboratory work, wrote and revised the article. Mohamed Ibrahim Abu Hassan, Hazlina Abdul

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Ghani, and Nor Wati Nur Atikah Mustafa designed the research, supervised the research work, edited, and approved the article submission.

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