

# Preparation and evaluation of particle size and its distribution in a resin composite prepared by dental amalgam powder incorporated in a bonding agent through scanning electron microscope

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## Abstract

**Introduction:** Dental resin composite is made up of a matrix reinforced with fillers. Different fillers are used for the preparation of resin composite. Dental amalgam powder incorporation in a resin devoid of fillers and its evaluation through scanning electron microscope (SEM) was performed in this study. Evaluation of filler particle size and their distribution through SEM in a resin composite prepared by mixing a bonding agent and dental amalgam powder.

**Material and Methods:** Commercially available bonding agent and dental amalgam powder were mechanically mixed through an amalgamator in a ratio of 20%. Particle size was evaluated before and after mixing and particle distribution in the resin composite matrix was evaluated through SEM. Micrograph measurements were performed through Adobe Acrobat Reader software (version 10).

**Results:** Mean values of particles were 15.15 $\mu$ m for dental amalgam powder. Which when mixed with bonding agent was 12.14 $\mu$ m. Particle distribution on the basis of size after mixing with resin was found non-significant. Paired samples two tailed T- test when applied result was 0.14 through SPSS version 16 ( $p \leq 0.05$ ).

**Conclusions:** An even filler particle distribution of the resin composite was confirmed. Dental amalgam powder particles as filler in a bonding agent can be used to produce a resin composite. Dental amalgam powder is a potential contender to be substituted as filler for dental resin composites for posterior teeth. It can also be used when mixed with a bonding agent for lining, basing, luting and bonding of orthodontic brackets etc., as an intermediate base which may provide an inherent antiseptic environment.

**Keywords:** Scanning electron microscopy; dental resin composite; metallic composite.

## Introduction

Resin composite for dental restorations are used since long. It consists of a matrix phase and a dispersion phase with an intermediate phase which is considered as a coupling phase.<sup>1</sup> Dispersion phase comprises of fillers particles of different sizes and shapes. This phase provides strength and bulk of the resin composite restorative

material. Physical and especially mechanical properties of a resin composite restorative material are dependent on their filler particles. Two main disadvantages associated with resin composites are polymerization shrinkage and degree of conversion which can be manipulated through filler content.<sup>2,3</sup> Different fillers have been tried to be incorporated in different proportions and assorted shapes and morphology and has been a hot topic for dental resin composites.<sup>4,5</sup> Latest bonding agents are considered as multi-functional and are claimed and proved to act as adhesive for all the materials such as dentine, enamel, amalgam, steel and ceramic etc.<sup>6</sup>

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Bonding agents are devoid of fillers. Introducing fillers in it will result in a complete resin composite restorative material. Routinely used monomers are Bisphenol-A glycidyl methacrylate (bis-GMA) and TEGDMA (Tri ethylene glycol di methacrylate) usually in a concentration of 60% and 40% respectively, which is mixed in laboratory. Evaluation of resin and metal bonding has been done previously with success.<sup>7</sup> Metal particles as fillers were previously used in different studies.<sup>8,9</sup> Lathe cut dental amalgam powder was considered as of diverse shape and size even if it is of fine grain particles. The size of these particles may be upto 35-50 $\mu\text{m}$ .

In this study particle size and thorough mixing of commercially available dental amalgam powder as filler in a commercially available bonding agent were evaluated. Materials were mixed mechanically through an amalgamator. Evaluation of fillers incorporated in dental resin composites with scanning electron microscope has not been done till now.

Hypothesis for the study was even distribution of filler particles can be accomplished through amalgamator for dental composites.

## Material and Methods

A commercial preparation of bonding agent BC-Plus (Vericom Co., Ltd. Korea) was used. Fillers used were also commercially available dental amalgam powder DENTOFIL-HC (Amin sons. Pakistan). Dental amalgam powder was treated ultrasonically (ULTRASONIC CLEANER VGT-2000 Digital. China) for three minutes in acetone to dissolve any organic material bonded during manipulation or handling of material. Material was heated in an electric oven at 37 $^{\circ}$  C for 24 hours for removal of acetone from amalgam particles.

The powder was weighed on an electric balance with high precision (Figure.1). An empty capsule previously cleaned was placed

and weighed four grams of dental amalgam powder transferred to it. Lights were turned off and carefully one gram bonding agent was added to it for obtaining an 80% powder to 20% liquid ratio. The capsule was tightly packed with Teflon tape and then mixing was done through amalgamator (YDM Amalgamator Hangzhou Yinya New Materials Co., Ltd. China.) at low speed for 10 seconds. After mixing Teflon tape was removed carefully

Stubs (Figure.2) were prepared through placing a non-conductor double tape upon which amalgam powder particles were sprinkled. Excess material was removed by shaking the stub holding upside downwards. Amalgam particles incorporated bonding agent was applied to another stub and light cured immediately for 10 seconds. The cured material was roughened with 1000 grit silicon carbide abrasive paper. Gold sputtering was performed by placing stubs in vacuum and then stubs were placed in the chamber of electron micro scope. Scanning electron microscopy was performed at magnification X350 with a 20 kV current (JSM 5910; Jeol, Japan) and micrographs were obtained (Figure.3&4).

## Results

Scattered particles of dental amalgam powder with diverse shapes were found in SEM study. Size of particles mainly ranges from 35-50 $\mu\text{m}$  (Table I) with agglomerates when seen at X350 magnification at 20 kV.

After mechanical mixing and curing, filler particles were thoroughly distributed in the matrix. Almost no agglomerates were found in different areas under focus. Some air bubbles were also present while on the other hand, some areas were found to be not affected by the silicon carbide paper.

was found 15.15 $\mu\text{m}$  with a standard deviation of 7.61 $\mu\text{m}$ . While after mixing it was 12.14 $\mu\text{m}$  with a standard deviation of 5.12 $\mu\text{m}$  (Table II). The mean difference in particle size before and after mixing was 3.00 with a standard

deviation of 10.47 (Table III). Readings obtained from SEM calculated by using adobe reader software (version 10) which provided values in mm. Millimeters converted into



Figure 1: High precision scale.



Figure 2: Prepared stubs

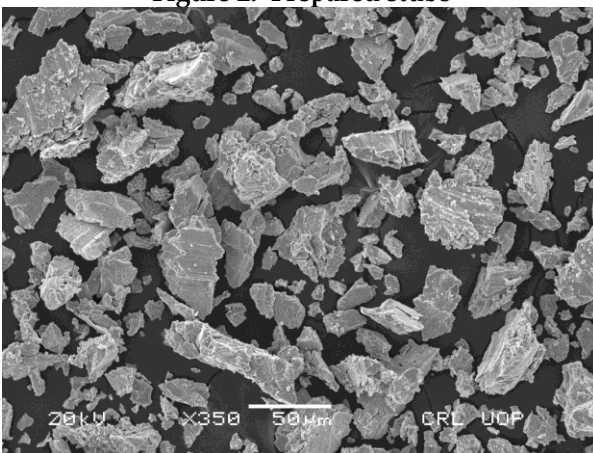


Figure 3: Dental amalgam powder through SEM.

micro meters by dividing 50 by 17.61 a value of 2.83 obtained which was multiplied to obtained the actual value.

Two tailed T test provided a value of 0.14 which is more than p value 0.05 selected for the study which indicates that there is a uniform distribution of particles before and after mixing with bonding agent (Table III).

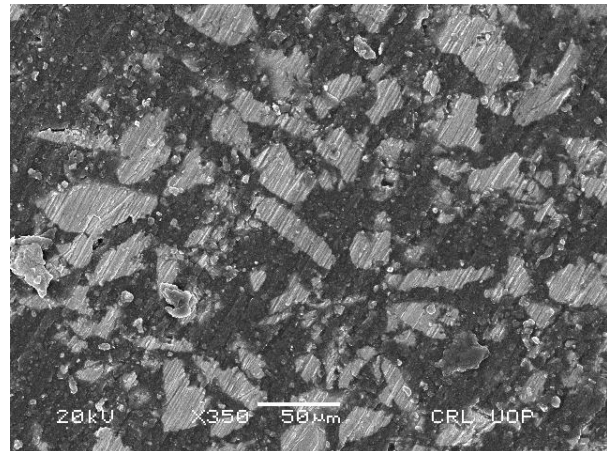


Figure 4: Dental amalgam powder mixed with bonding agent.

## Discussion

Different particle sizes in manufacturing of resin composites were observed and used in experimental and commercially available resin composites.<sup>10</sup> Aimed to produce better mechanical properties same approach was followed in the present study. A commercially available material having different particle size commonly used in dentistry was dental amalgam powder. Mercury was a great concern, so pre-amalgamated dental amalgam powder was not used to exclude the friction of mercury used in pre-amalgamated type. Spherical type of dental amalgam powder was also not used because of homogeneity of particle shape though the sizes may differ. Diversity in lathe cut powder for its various shapes provides a good footage for its selection as filler for resin composite. Different sized particles also helps in load transfer from resin matrix to fillers and prevents slip plane formation due to the

hindrance of bigger particles from smaller and those which attained an interstitial space between these particles. All the above mentioned phenomenon was accomplished through the use of dental amalgam powder due to its heterogeneity.<sup>11</sup>

**Table I: Measurements of particle size before and after mixing**

No	Particle size before mixing ( $\mu\text{m}$ )	Particle size after mixing ( $\mu\text{m}$ )
1	22.87	9.70
2	23.08	14.37
3	10.53	12.31
4	14.99	13.21
5	13.37	10.47
6	28.79	9.73
7	36.25	9.70
8	13.74	12.39
9	13.40	8.20
10	9.45	23.60
11	8.88	16.58
12	9.76	10.38
13	16.46	6.33
14	19.84	10.61
15	10.47	14.74
16	19.50	14.60
17	10.42	7.52
18	24.78	7.21
19	4.74	13.49
20	5.96	13.95
21	16.32	6.56
22	8.06	9.02
23	20.15	7.92
24	6.72	6.50
25	16.95	23.06
26	6.92	24.16
27	9.82	17.29
28	23.59	6.50

**Table II: Mean, standard deviation, minimum and maximum values of particle size through SEM**

		Particle Size Before Mixing ( $\mu\text{m}$ )	Particle Size After Mixing ( $\mu\text{m}$ )
N	Valid	28	28
	Missing	0	0
Mean		15.1554	12.1464
Std. Deviation		7.61065	5.12287
Minimum		4.72	6.33
Maximum		36.13	24.16

Traditional resin composites comprise of fillers of particle size up to 30  $\mu\text{m}$ .<sup>12</sup> Size of fillers may vary, it was used up to 60 to 80 $\mu\text{m}$

in length and 100 $\mu\text{m}$  in diameter.<sup>13</sup> Particle size of 35-50 $\mu\text{m}$  was usually found in dental amalgams. Average particle size evaluated through SEM in current study before mixing with bonding agent was found 15.21 $\mu\text{m}$  as the mean values were calculated. After mixing it was recorded on the basis of their mean value as 12.14  $\mu\text{m}$ . These values confirm the particle size values corresponding to densified resin composites particle size, which are considered best for posterior restorations.<sup>14</sup>

**Table III: Paired sample T test before and after mixing with bonding agent**

Paired Samples Test			Pair 1
			Before Mixing - After Mixing
Paired Differences	Mean		3.00893
	Std. Deviation		10.47207
	Std. Error Mean		1.97904
	95% Confidence Interval of the Difference	Lower	-1.05172
		Upper	7.06957
T		1.52	
df		27	
Sig. (2-tailed)		0.14	

Bonding agents provide a good seal for metals as in case of bonding between amalgam filling materials and metallic or even ceramic brackets used in orthodontics therefore, these agents were used for mixing of silver amalgam particles as described earlier.

Reason behind using commercially available material is to take advantage from the commercial techniques and standardization which cannot be achieved in experimental procedures. It also saves the time and providing maximum attention towards our research. Commercial preparation had more relevant and accurate clinical approach with tested and quality ensured and controlled results.

Differences in mean values of particle size correspond to the mean values of particles present in the dental amalgam powder. The great diversity in the particle size depicts its variation even in its statistical analysis.

Other means of evaluation of particle size were X-ray microanalysis and granulometry.<sup>15</sup> Energy-dispersive X-ray spectrometry was also an alternative for evaluation of particle size. Profilometry analysis is another means of evaluation, which is used for surface roughness for resin composite materials. Tactile profilometry is the most common method used for resin composite material determination.<sup>16</sup> These are difficult and time consuming procedures. Their accuracy is not as reliable as SEM. There are a number of confounders, which can modify the results associated with these methods. Evaluation through SEM is a standard protocol followed by researchers in their studies.<sup>17,18</sup>

Agglomerates of particles were found in powder form which was consistent with other study performed by Wan et al<sup>17</sup> while on mixing with resin these were not found and an even distribution was obtained without any gaps present in between particles and resin matrix. This is a strong evidence of even distribution of particles within the resin matrix without using any coupling agent.

Fillers play a definite role in the clinical success of resin composites. Different blends of different particle size and shapes were used in different studies. A blend of different shape and size when used in a resin composite is usually called a hybrid resin composite. Currently, hybrid resin composites are used substantially in clinical practice. Hybrid resin composites were considered as a gold standard in posterior resin composites.<sup>19</sup> This study was a proceeding towards a hybrid resin composite formulation.

Incorporation of copper and silver containing material in a composite has a double impact. It improves its strength as a filler and both of

these elements have anti-microbial properties.<sup>20,21</sup> These aspects of silver and copper turn the attention towards its implication in oral biology and orthodontic cementation of brackets. Oral environment specially interphase between dentine and filling material containing these elements is a point of interest. Till now there is no composite material with inherent anti-microbial properties. Though some elements have been incorporated experimentally.<sup>22,23,24</sup>

## Conclusions

Particle size evaluated and even filler particle distribution of the resin composite was achieved and confirmed through SEM.

## Recommendations

Further trials are needed to evaluate different aspects of this newly tested composition.

Dental amalgam powder can be substituted as a filler for posterior dental resin composites.

It can also be used when mixed with a bonding agent in anterior teeth as an intermediate base (layer) which may provide an inherent antiseptic environment.

## Conflict of interest

Benefits or any other favor were not taken nor demanded through this study from any institution or person.

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