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Research Article

Evaluation of three physical mixing methods of nanoparticles to orthodontic primer

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Abstract: Background: Demineralization and white spot lesions are the most common complications in fixed orthodontic treatment. It is useful to enhance the remineralization properties of the orthodontic primer by the addition of remineralizing agents. Fluoride and calcium are regarded as the main component of enamel fluorohydroxyapatite crystals. This pilot study compared three mixing methods of calcium fluoride nanoparticles ($nCaF_2$) with conventional orthodontic primer (Transbond XT™) to develop a primer with enamel remineralization properties. Materials and methods: The nanoparticles were added to Transbond XT™ primer to form 20% (w/w) of the final solution. Three dark plastic bottles were prepared and stored until mixing. The first sample was mixed by a Vortex machine, the second was mixed with an electric agitator, and the third one was mixed with a customized plastic spatula adapted to a dental engine and a straight handpiece. Cured blocks of the developed primer were prepared and were examined for homogeneity, cracks, and agglomeration of the nanoparticles within the primer using Field Emission Scanning Electron Microscopy (FESM). Results: The third Sample showed a continuous distribution of nanoparticles with no apparent cracks or agglomeration of nanoparticles. In contrast, a higher agglomeration was seen in the first sample than in the other two. Conclusion: Mixing of $nCaF_2$ with Transbond XT™ orthodontic primer was best achieved by the customized plastic spatula adapted to a dental engine compared to Vortex and electric agitator machines.

Keywords: calcium fluoride, nanoparticles, orthodontic primer.

Introduction

Nanoparticles are used to raise the benefits of the resultant nanocomposite materials in many fields like photonic, energetic, electronic, and biomedical materials [1]. All applications require proper handling and mixing to get the best properties. The unique effects of nanoparticles emerge from their size reduction. Moreover, a larger surface area per unit volume will be achieved when a particle decreases to the nano-size (1–100 nm). More importantly, enriched numbers of molecules or atoms per the same area of the identical particles of larger size [1, 2]. The mixing quality is examined by analyzing high quality and magnified images of the particles within a mixture sample utilizing microscopy. The photos of the mixture of particles can be distinguished by a particular particle shape, color, or some other surface characteristic [3, 4].

Testing homogeneity and agglomeration of nanoparticles in the primer requires high-resolution and magnified images to distinguish the characteristics of the nanoscale. A variety of electron microscopes and energy dispersive X-ray spectroscopy can be utilized. Moreover, atomic force microscopy can provide a topographical map of the surface of the sample with a resolution explaining a few nanometers with the ability to distinguish between different nanoparticles if two or more nanoparticles were mixed in the same sample. Scanning electron microscope (SEM) imaging may be more straightforward and reliable if only a qualitative assessment of mixing is required [4, 5].

SEM is an electron microscope that can image a sample by scanning with a focused beam of electrons. The electrons will interact with the atoms of the sample, then signals that give information about the topography and composition of the sample will be produced. In SEM, secondary electrons are emitted by exciting atoms by the primary electron beam and then detected by the secondary electron detector. The secondary electrons numbers that can be detected depend on specimen topography and contents [5].

Calcium fluoride is considered a calcium and fluoride reservoir in the oral cavity; this material will be dissolved when pH drops, releasing fluoride and calcium in the oral cavity [6]. Fluoride and calcium are the main components to form fluorapatite crystals after the remineralization process following elevation in pH in the oral cavity [7]. Furthermore, when calcium fluoride particles are used in nano size, they will have more effect than their larger particles counterparts as they will have a larger surface area and more fluoride and calcium molecules released. That will make $nCaF_2$ a good choice for caries prevention [8].

Incorporating nanoparticles into liquid is particularly challenging due to the powerful attractive forces between the nanoparticle. The degree of difficulty varies in different applications[4]. It might be dictated by particle shape and size, fluid type, and the existence of the dispersing materials; different nanoparticles require different shear rates for deagglomeration for mixing[9]. Nevertheless, turning to more intensive devices is advised when traditional mixers fail to achieve the preferred dispersion level[10]. Different mechanical methods were used to mix nanoparticles in liquid or gel solutions, like using an electric agitator shaker[11], a Vortex machine [12], and a dental handpiece with a plastic spatula[13].

This pilot study aimed to evaluate three physical mixing methods of $nCaF_2$ in conventional orthodontic primer to achieve $nCaF_2$ containing primer with higher quality regarding homogeneity, crack-free, and agglomeration using FESEM.

Materials and methods

$nCaF_2$ powder was purchased from Nanoshell Company (USA, code:7789-75-5) with 99.5% purity of 80-100 nanometers size, crystalline structure form, 1400˚C melting point, and 2500˚C boiling point. Transbond XT™ orthodontic primers (3M-Unitek, Monrovia, USA) were used as the conventional primer where $nCaF_2$ will be added. The primer and the nanoparticles were weighed using a highly sensitive four digits balance (Satorius company, Germany). The powdered nanoparticles were added to Transbond XT™ primer 20% as a weight percentage. Three dark plastic bottles were prepared and stored until mixing procedures were performed. Each primer bottle was used for different mixing methods as follows:

1. V sample: mixing was performed using a Vortex machine (Huma Twist, Wiesbaden, Germany) (Fig. 1 A) for about 2 minutes [11].

2. E sample: mixing was achieved by a shaking method using an electric agitator shaker (Fig. 1 B) (Vortexmixer, China) at three intervals of 15 seconds each. The first 15 seconds was at a speed of 800/min with a spiral motion; the second was at a speed of 1800 /min with vibration motion. The third at a speed of 800/min at a spiral agitation motion[12].

3. D sample: $nCaF_2$ were mixed with primer using a customized plastic spatula adapted to a dental engine and a straight handpiece (Fig. 1 C); different speeds were used for three minutes to get adequate homogeneity.

The mixing process was accomplished in a darkroom. Each prepared primer was cured in 8 mm long and 2 mm thick blocks using a dental light curing device and then sent to FESEM examination, in addition to a block of a control primer (without adding nanoparticles). The distribution of fillers within the original primer was examined using FESEM in 50, 100, and 200 KX to assess the homogeneity, cracks, and agglomeration of tested primers prepared in this study.

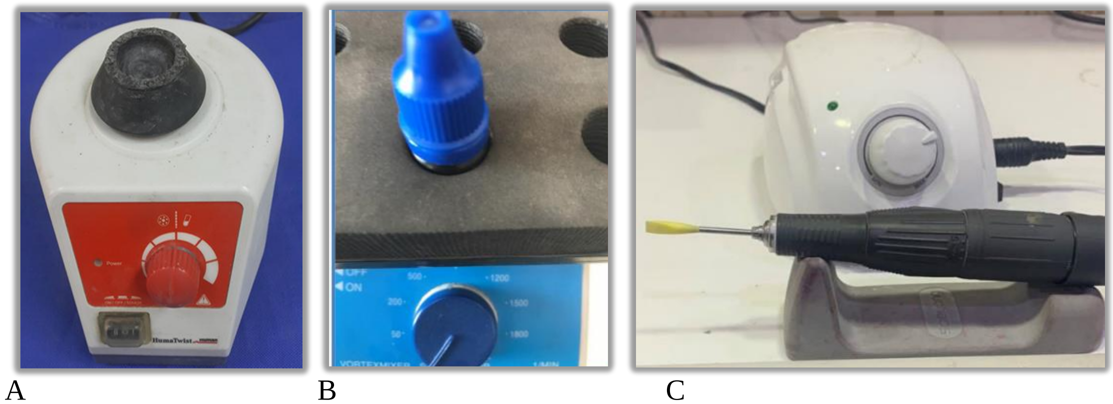

Figure 1: The three methods that were used to mix the nanoparticles in orthodontic primer. A: Vortex machine, B: electric agitator shaker, C: customized plastic spatula adapted to a dental engine and a straight handpiece

Results

FESEM was performed on the control primer (Fig. 2), and the three developed primers with different mixing methods of $nCaF_2$ to the control primer. The result of the V sample revealed minimum cracks with a continuous distribution of the nanoparticles but with the presence of a large area of agglomerated nanoparticles (Fig. 3). However, the E sample image seemed to have minimum cracks and multiple agglomeration areas (Fig. 4).

Finally, the D sample image showed a continuous distribution of nanoparticles with no apparent cracks or agglomeration within the primer (Fig. 5).
The evaluation of the SEM images was performed and then validated by an expert in SEM image investigation.

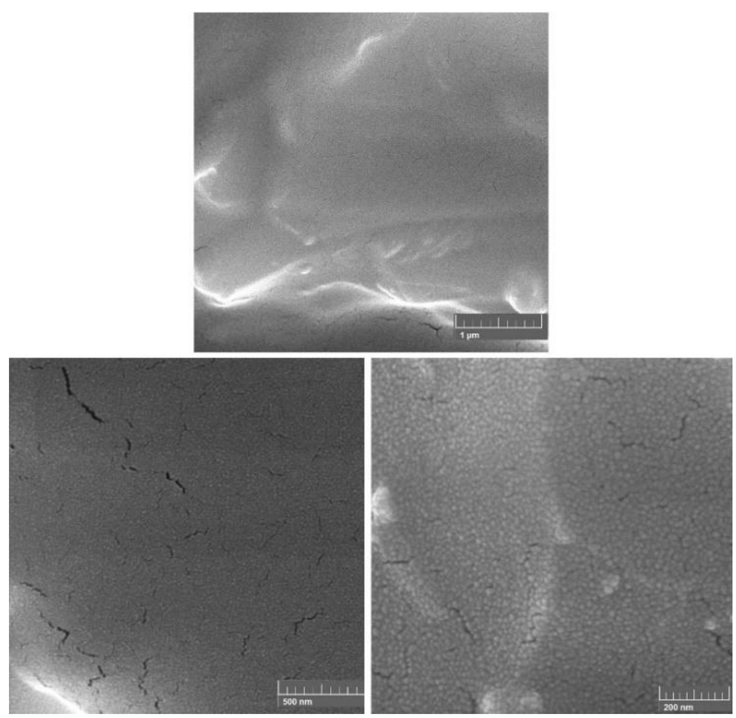

Figure 2: The scanning electron microscope image for control primer (Transbond XT™) magnified by 50 KX (Upper) 100 KX (lower right), and 200 KX (lower left)

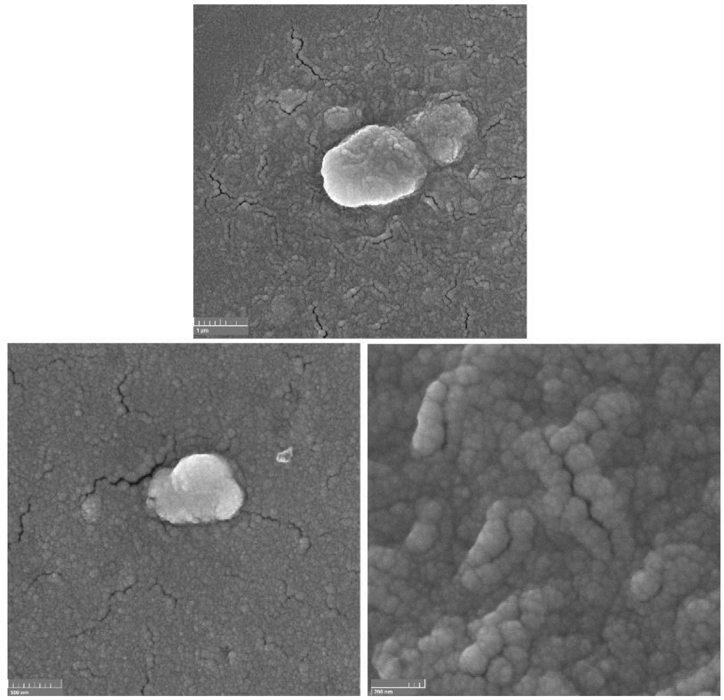

Figure 3: The scanning electron microscope image for prepared primed mixed by vortex machine magnified by 50 KX (Upper) 100 KX (lower right), and 200 KX (lower left). Figures showed good distribution of nanoparticles with minimum cracks and an abundant area of agglomerated nanoparticles.

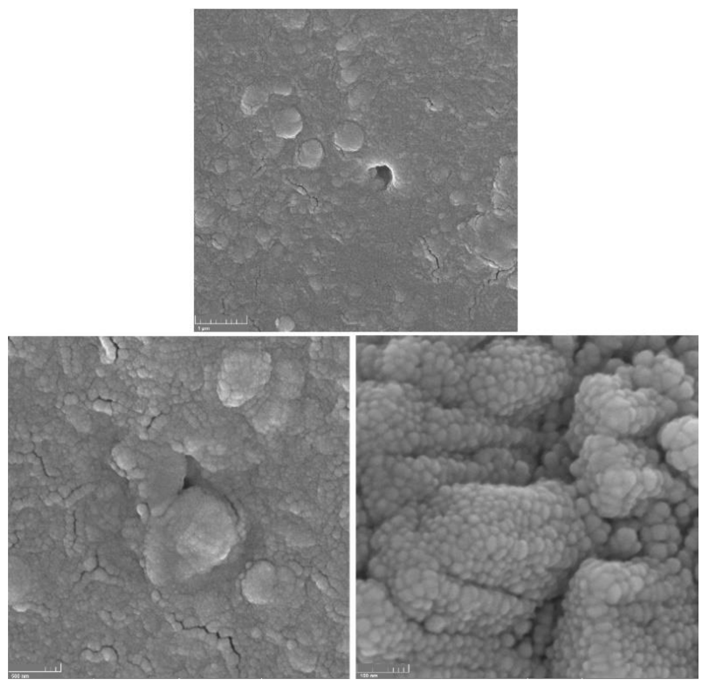

Figure 4: The scanning electron microscope image for prepared primed mixed by mixed using vortex machine magnified by 50 KX (Upper) 100 KX (lower right), and 200 KX (lower left). Figures showed minimum cracks and multiple areas of $nCaF_2$ agglomeration

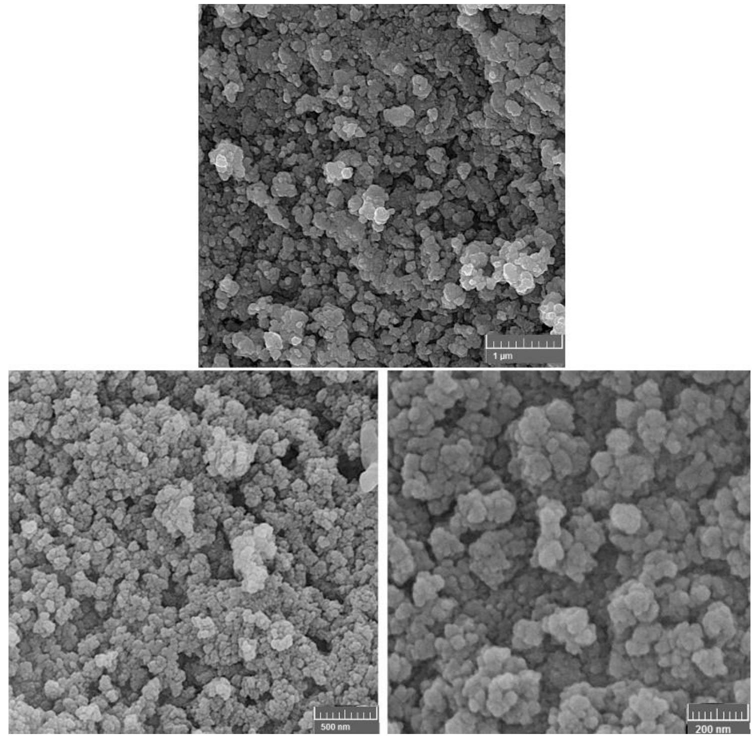

Figure 5: The scanning electron microscope image for prepared primed mixed by mixed using a customized plastic spatula adapted to a dental engine and a straight handpiece magnified by 50 KX (Upper) 100 KX (lower right), and 200 KX (lower left). Figures showed a better distribution of nanoparticles with no cracks and no significant agglomeration

Discussion

Many researches included the addition of remineralization and antibacterial agents to dental adhesives to antagonize bacterial colonization and promoted enamel remineralization. They found that the additives of nano-sized particles could enhance mechanical characteristics compared to the microsized material of same chemical structure [14, 15].

Moreover, the large surface area of the materials with nanosize in addition to the presence of unbounded atoms makes more reactivity of the material in compared to the same materials in macro size [16].

Nanoparticles can be added or mixed to resin matrix or other materials by salinization [17] or by mechanical method. In our study, $nCaF_2$ was not salinized and only physically incorporated in the resin matrix [11, 12, 13]. The idea beyond that is to make calcium fluoride nanoparticles free (not bonded chemically to orthodontic primer). This would make these particles easily dissolvable when the primer layers disintegrate after exposure to acid attacks in the patient mouth. Moreover, $nCaF_2$, when added to low viscous liquid (primer), will penetrate as deep as the acid etch conditions the enamel surface and consequently render the enamel surface surrounding the orthodontic bracket more resistant WSLs and enhance the remineralization process.

In the current study, vortex machine mixing revealed a mixing with poor homogeneity and significant nanoparticles agglomerations, which might be due to the design of the device, which is indicated mainly for mixing small vials of liquid that might not be intensive enough to mix nanoparticles within the primer liquid [18]. Furthermore, the main mixing motion in a vortex machine is circular. This motion is transmitted from the device to the liquid via the manual touching of the plastic primer container to the rubber cap on the top of the device. This motion might not be enough to deagglomerate the nanoparticles within the primer liquid.

The electric agitator mixing showed better homogeneity than vortex machine mixing; however, multiple agglomerations could be seen in the FESEM image. This area of agglomeration can be explained by the force of this device which was not enough to break down attractive forces between the nanoparticles as this deceive manufactured in a way that prevents excessive heating of the laboratory solution, especially the microbial culturing solution [9]. Better homogeneity and less agglomeration were shown in the dental engine group. The different speeds and the time (three minutes) might be suitable for getting enough shaking motion to break down agglomerates and better distribution of the nanoparticles to get adequate homogeneity. On the other hand, the heat produced by dental engine mixing is not of clinical importance regarding dental primer and composite materials [19, 20, 21].

The efficacy of mixing methods may not produce the same results if different materials or particles size is used. So, future studies are recommended to test the mixing method's effectiveness using different nanoparticles that can add bioactive and preventive properties to orthodontic primers. Moreover, different types of dental primers with different viscosity can be involved.

Conclusion

Mixing $nCaF_2$ with orthodontic primer liquid showed better homogeneity of $nCaF_2$ with minimal agglomeration and cracks when mixed with customized plastic spatula adapted to a dental engine and a straight handpiece as compared to mixing by vortex or electric agitator machines. Future testing using other nanoparticles and/or other conventional dental primers is recommended.

Conflict of interest

The authors have no conflicts of interest to declare.

Author contributions

JYD., BH. and WA. contributed to the design and implementation of the research, to the analysis of the results and to the writing of the manuscript

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Ethical approval

All of the individuals were given thorough information about the study and the procedures involved, and their informed consent was acquired on a form approved by the ethics committee of the University Of Baghdad \ College Of Dentistry.

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تقييم ثلاث طرق للخلط الفيزيائي للجسيمات النانوية مع بادئ لاصق تقويم الأسنان: دراسة تجريبية

رؤوف طعمه, ياسر عبدالكاظم, كرانت ماكنتاير

المستخلص

الخلفية: ان تحلل مينا الاسنان وآفات البقع البيضاء هي المضاعفات الأكثر شيوعًا في علاج تقويم الأسنان الثابت. من المفيد تعزيز خصائص إعادة البناء لطبقة المينا عن طريق إضافة عوامل إعادة بناء طبقة الاسنان الخارجية و يعتبر الفلوريد والكالسيوم المكون الرئيسي لبلورات هيدروكسيباتيت المكونة للاسنان. قارنت هذه الدراسة التجريبية ثلاث طرق لخلط الجسيمات النانوية لفلوريد الكالسيوم لتطوير بادئ لصق تقويم الاسنان بخصائص إعادة تكوين طبقة المينا. المواد والطرق: تمت إضافة الجسيمات النانوية لفلوريد الكالسيوم بنسبة 20٪ كنسبة مئوية من الوزن الى بادئ لاصق

التقويم المسمى (Transbond XT™) . تم تحضير ثلاث قوارير بلاستيكية داكنة اللون وتخزينها لحين إجراء عملية الخلط . تم خلط العينة الأولى بآلة Vortex (عينة V)، وتم خلط العينة الثانية بالخلاط الكهربائي المختبري (عينة E) ، بينما تم خلط العينة الثالثة بملعقة بلاستيكية مصنعة خصيصا كي تتكيف مع محرك أسنان المختبري وقبضته المستقيمة (عينة D) .بعد ذلك تم تحضير عينات مصلبة ضوئيا للبادئ المطور. تم فحص العينات من أجل التجانس ، والشقوق ، وتكتل الجسيمات النانوية داخل العينات المصلبة باستخدام المجهر الإلكتروني (FESM).

النتائج: أظهرت صور المجهر الالكلتروني للعينة (D) توزعًا جيدا للجسيمات النانوية مع عدم وجود تشققات ظاهرة أو تكتل للجسيمات النانوية داخل العينة. في المقابل لوحظ تكتل أعلى في العينة (V)مقارنة بالعينتين الأخريين. الاستنتاج: تم الكشف عن أفضل نتيجة لمزج الجسيمات النانوية لفلوريد الكالسيوم مع بادئ لصق التقويم بواسطة ملعقة بلاستيكية مصنعة خصيصا كي تتكيف مع محرك أسنان المختبري وقبضته المستقيمة مقارنة بالخلط بآلات Vortex و الخلاط الكهربائي المختبري