Powder Delivery in Dental Restoration Rapid Prototyping Process

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ABSTRACT

Rapid prototyping of dental restoration has been investigated for its potential to save time and cost. In this paper, a powder delivery system was developed to deliver dental porcelain powder accurately into 2-D shapes for dental restoration. Aqueous suspension of the porcelain was prepared as the vehicle for the powder delivery. An integrated dry and wet ball-milling process was developed to reduce the particle size and minimize agglomeration. The reduction in particle size and minimization of agglomeration increased the suspension stability. The optimization of the pH value of the suspension also provided an additional means to achieve the stability and reduce the viscosity of the suspension. With the optimization of the particle size, pH value and solid concentration in the suspension, desired powder shapes were successfully delivered.

Key word: Rapid prototyping, dental restoration, powder delivery, ball milling.

INTRODUCTION

There are currently more than 10,000 dental laboratories in the US and a majority of these laboratories use porcelain-fused-to-metal (PFM) restoration for permanent fixed prosthodontics. PFM restoration is a very time consuming and labor intensive work because PFM restoration requires a multi-stage process using multiple materials (both ceramics and metals) and each stage involves multiple processing steps [1]. For example, it normally takes 2-4 weeks to make a three-unit bridge and labor costs account for about 90% of the final cost. This study is part of an overall effort to develop a novel multi-materials laser densification (MMLD) process for dental restorations [1-3]. This process utilizes laser-assisted solid freeform fabrication (SFF) to fabricate artificial dental units layer-by-layer directly from a computer model without part-specific tooling and human intervention. As such, the labor cost will be substantially reduced, and better and faster dental restorations will be achieved.

One of the key issues in the MMLD process is to deliver dental alloy and porcelain powders to the desired locations precisely and accurately point by point, line by line and layer by layer. Point-by-point and line-by-line powder deliveries are essential because there are multiple materials involved in a single plane for dental restorations. This study focuses on how to delivery dental porcelain powder precisely and accurately to the desirable locations in point, line and

layer forms before laser densification of these delivered powder shapes. Powder suspension and paste have been selected as the vehicles to deliver dental powders because wet suspensions and pastes provide a better control on the shape of the powder compact delivered than a dry powder does. Effects of the particle size, the pH value of the suspension and the concentration of solids in the suspension on powder delivery have been investigated. The results are presented below.

EXPERIMENTAL

The dental porcelain powder was provided by Degussa-Ney Dental Inc., Bloomfield, CT. The chemical composition of the porcelain is confidential; however, it is within 5% of the nominal composition of the Weinstein patent [4], which has the following composition (wt%): 63.40% SiO_2, 16.70% Al_2O_3, 1.50% CaO, 0.80% MgO, 3.41% Na_2O, and 14.19% K_2O. The typical morphology and microstructure of the porcelain powder are shown in Figure 1. It can be seen that the as-received powder has angular shapes and their equivalent particle sizes range from 1 to 50 micrometers. To reduce the size of the porcelain powder, ball-milling process was conducted using a SPEX 8000 mill machine. The vial and balls for the ball milling process were all made of alumina, and thus the alumina concentration in the milled powder could be slightly higher than that of the original powder should wear of the alumina vial and balls take place during the milling. The weight ratio of the powder to alumina balls was 2.5 in all of the ball milling experiments.

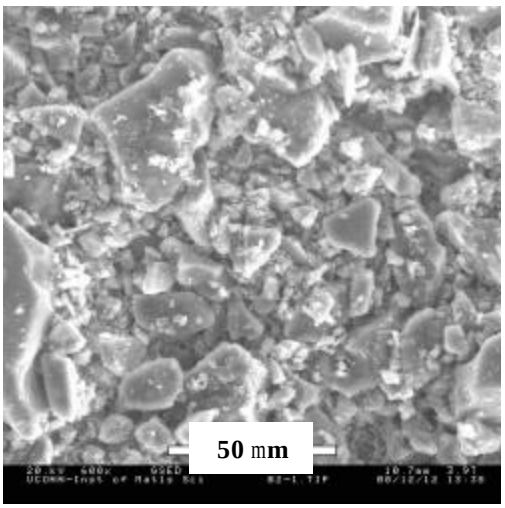

Fig. 1 ESEM image of the as-received dental porcelain powder.

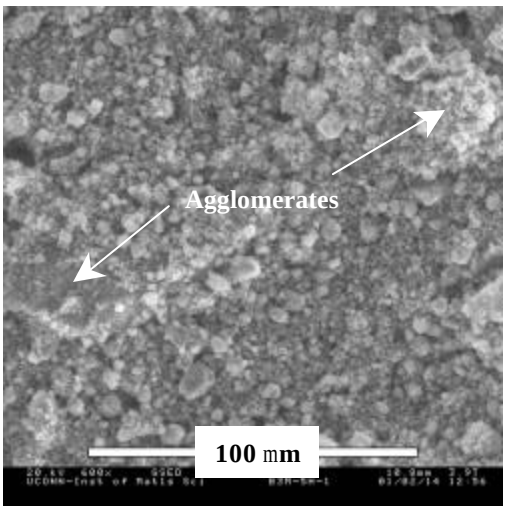

Fig. 2 ESEM image of the porcelain powder after 5 hours of dry ball milling.

Both the as-received and milled powders were used to prepare suspensions with de-ionized water as the liquid media. The suspensions prepared were subjected to the viscosity measurement and suspension delivery evaluation. The porcelain suspension was delivered using a dental restoration machine made in our laboratory. The machine contains a X-Y-Z table controlled by a computer through a DMC-1800 Galil Motion control card. The suspension was held in a barrel at the end of which there was a nozzle for suspension delivery. The diameter of the nozzle can be changed for the evaluation of the nozzle size effect. The sizes of the delivered powder suspension points and lines were measured and compared to the nozzle diameter.

Both the as-received and milled porcelain powders were characterized using an environmental scanning electron microscope (Philips ESEM 2020) to obtain the morphology and size of the powders. Analysis of the specific surface area of the powder was conducted using nitrogen adsorption based on the Braunauer-Emmett-Teller (BET) theory (Quantachrome NOVA 1000). The viscosity of the porcelain suspension was determined using a Brookfield DV II digital viscometer. The zeta potential of the suspension was measured in the National Science Foundation Engineering Research Center for Particle Science and Technology at the University of Florida using a Zeta-Reader instrument.

RESULTS AND DISCUSSION

1. Effect of Ball-Milling Process on Porcelain Powder

As shown in Figure 1, the as-received porcelain particles are substantially larger than 1 μm and thus not suitable for preparing stable colloidal suspensions. To achieve a stable colloidal suspension, the size of solid particles normally needs to be smaller than or about 1 μm. Thus, ball milling was investigated in this study for its potential to reduce the porcelain powder size. Figure 2 shows the ESEM image of the porcelain powder after 5 hours of dry ball milling. When compared with the as-received powder, the size of porcelain particles has been significantly reduced by the ball milling process. After 5 hours of dry milling most of the particles are smaller than 5 μm, whereas 20 μm particles are very common in the as-received powder. The specific surface area of the porcelain powder as a function of the time of dry milling is shown in Figure 3. As expected, the specific surface area of the porcelain powder increases with milling time. At 8 hours of milling, the specific surface area has increased 8 times over the as-received powder, suggesting that the average particle size has been reduced to about 1/3 of the original.

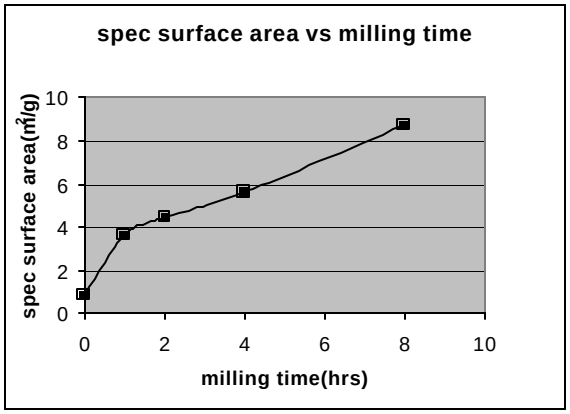

Fig. 3 The specific surface area of the porcelain powder as a function of dry milling time.

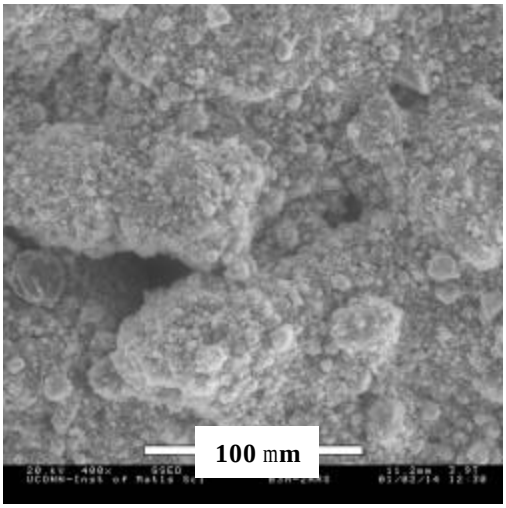

Fig. 4 Particle agglomeration in the porcelain powder with 2 hours of dry milling.

It is also noted from Figure 3 that the increase rate of the specific surface area becomes smaller after 1.5 hours of milling. Detailed SEM examination of the milled powder reveals that the reduced rate for increasing the specific surface area is related to powder agglomeration

during ball milling. Powder agglomeration will degrade the efficiency of the comminution process and is highly undesirable. It is also found that powder agglomeration becomes obvious at about 2 hours of milling, coinciding with the time for changing the increase rate of the specific surface area shown in Figure 3. A typical morphology of powder agglomeration observed in the powder with 2 hours of dry milling is shown in Figure 4. Clearly, to improve the milling efficiency powder agglomeration during milling should be avoided.

In order to overcome the agglomeration problem and obtain a well-dispersed suspension, wet ball-milling process was also investigated. De-ionized water was used as the liquid media for the wet milling process. Figure 5 shows the typical morphology of the porcelain powder after 5 hours of wet milling. It can be seen that there is no agglomeration; however, the particle size is larger than that of the powder dry milled for 5 hours (Figure 2). This is consistent with the well established phenomenon that wet milling is less efficient in particle size reduction than dry milling [5,6]. In order to utilize the advantages of both dry milling and wet milling, we have developed an integrated dry and wet milling process in which the porcelain powder is dry milled for several hours (e.g., 2 to 4 hours) and subsequently wet milled for 1 hour. In such integrated milling process, the dry milling at the early stage of the process reduces the particle size while the wet milling at the later stage of the process breaks down agglomerates and produces a well dispersed suspension. This expectation was confirmed by experiments. Furthermore, the suspension prepared from the integrated milling process was much more stable than without milling or with dry milling only because the suspension from the integrated milling process has fine particles and few or no agglomerates.

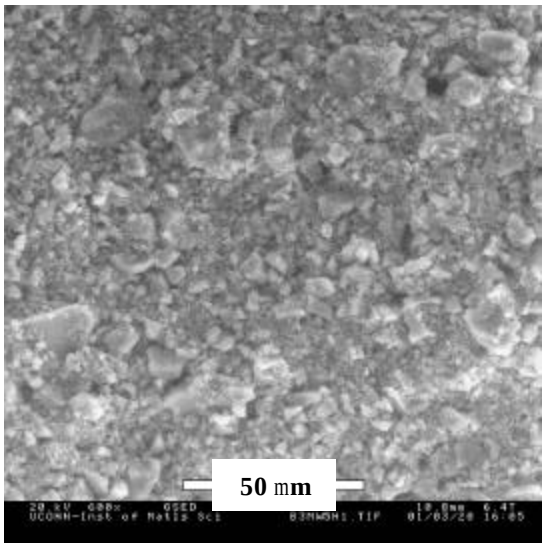

Fig. 5 Morphology of the porcelain powder after wet milling for 5 hours.

2. Influence of pH Value on the Stability and Viscosity of Porcelain Suspension

The stability of a colloidal suspension, according to the DLVO theory [7], is determined by the balance between the repulsive and attractive forces which particles experience as they approach. The repulsive force depends on the degree of double layer overlap. It has long been recognized that the zeta-potential is a good index of the magnitude of the repulsive interaction between particles. Figure 6 shows the zeta potential versus pH value curve of the porcelain

powder suspension. It indicates that the iso-electric point (i.e.p.) of the dental porcelain powder is at about 5.0. It is interesting to note that this number falls between the i.e.p. of Al_2O_3 and SiO_2. The i.e.p. of Al_2O_3 is at 8.7 [8], whereas SiO_2 at 2.0 [9]. Since Al_2O_3 and SiO_2 are the main components of the dental porcelain [4], the zeta-potential of the porcelain powder appears to be controlled by a combined interactive effect of these two compounds.

According to "Eilers and Korff Rule", the onset of instability of a suspension is associated with a rapid decrease in the value of the function ζ^2/κ(ζ: Zeta potential; κ: Debye-Huckel parameter). It can be derived from Figure 6 that between pH value 4 and 6, ζ^2/κ will have a rapid decrease in the value; thus, coagulation of the particles in the suspension will happen quickly in this pH range, leading to an unstable suspension. For suspensions with pH< 3 or pH> 6, the zeta potential of the porcelain powder changes slowly and the surface of powder particles is mainly positive charged (for pH< 3) or negative charged (for pH> 6), which results in relative large repulsive forces between particles and thus stable suspensions.

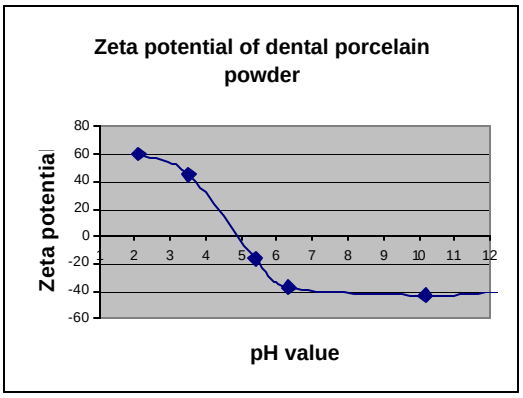

Fig. 6 Zeta potential of the dental porcelain powder as a function of pH value.

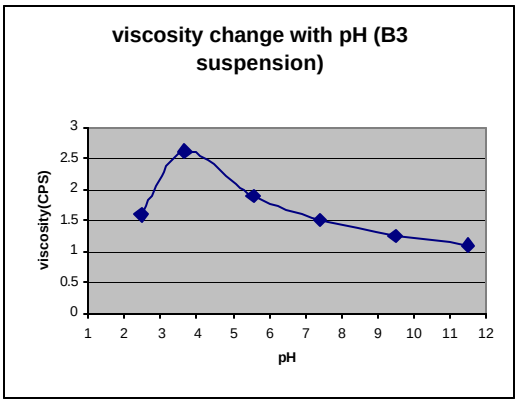

Fig. 7 Viscosity change of the porcelain suspension with pH value (8 vol% solid).

Measurement of the suspension viscosity was also consistent with the finding of the zeta potential measurement. Shown in Figure 7 is the viscosity of the 8 vol.% porcelain powder suspension as a function of the pH value. Note that the viscosity of the suspension peaks in pH range 3 – 6, consistent with the zeta potential curve that predicts the possible coagulation in pH range 3 – 6. Low viscosity of the suspension will translate into high flowability which makes the suspension easy to deal with in powder delivery process. Furthermore, the suspension with pH< 3 or pH> 6 has relatively good stability and thus a long shelf life. Since the suspension with pH range 7 – 10 has lower viscosities than the suspension of pH< 3, the suspension with pH range 7 – 10 has been selected for further powder delivery investigation. An additional advantage using the suspension with pH range 7 – 10 over pH< 3 is less corrosiveness offered by the suspension with pH range 7 – 10.

3. Delivery of Dental Porcelain Suspension

Delivery of dental porcelain suspension was done using an in-house designed machine that contains a X-Y-Z table and a barrel used for holding the suspension (Figure 8(a)). At the end of the barrel there is a nozzle the diameter of which can be changed for the evaluation of the nozzle size effect. The delivery of the suspension is controlled by combined effects of the surface tension and the gravity of the suspension. When the nozzle touches the substrate, the suspension

flows out of the nozzle; otherwise, the suspension will be held within the nozzle by the surface tension of the suspension. It was found that successful delivery of dental porcelain suspensions by the aforementioned machine and mechanism relied on the proper selection of the solid concentration and pH value of the suspension, the particle size of the powder, and the diameter of the nozzle. Large particles, small-diameter nozzles and high viscosity suspensions typically resulted in gradual blockage of the nozzle and eventually cessation of the suspension delivery process.

Figure 8(b) shows the porcelain suspension delivered in several 2-D shapes. The suspension shown in Figure 8(b) had a pH value of 10 and a solid loading of 20 vol.%. The diameter of the nozzle used was 0.5 mm. One of the determining factors for the dimensional accuracy of dental restoration is the accuracy of the suspension delivery which, in turn, is determined by the smallest point and line that can be delivered. The present study has revealed that the smallest point and line that can be delivered are affected by many factors. The key factors include the nozzle size, the viscosity and the flowability of the suspension. The smallest point that can be delivered now is 0.5 – 0.8 mm as showed in the middle and right bottom of Figure 8(b). On the upper left, two suspension lines were formed by delivering 30 individual points in each line with 30% overlap between the neighboring points. On the upper right, a square shape was formed by delivering 121 points in 11 lines with no overlap between the neighboring points. Thus, the width and thickness of a single line can be adjusted in the current delivery method by controlling the overlapping distance between the neighboring points.

Figure 8. (a) porcelain suspension delivery system, and (b) 2-D samples delivered.

CONCLUSIONS

A delivery process of dental porcelain powder has been developed using a suspension approach. The well-dispersed porcelain suspension is obtained by a newly developed milling process that integrates dry and wet milling processes. The dry milling at the early stage of the integrated process effectively reduces the particle size, whereas the wet milling at the later stage of the integrated process breaks down agglomerates and produces a stable suspension. The viscosity of the suspension strongly depends on the pH value of the suspension. Low viscosity suspensions are obtained when pH is about 10, which coincides with the pH value for obtaining stable suspensions as predicted by the zeta potential measurement. Low viscosity and stable

suspensions are beneficial for suspension delivery. This is especially true when very small points and lines are to be delivered. Without low viscosity and stable suspensions the nozzle for delivering suspensions will be gradually blocked and eventually cease to function. The smallest point delivered that has been achieved in this study is about 0.5 mm in diameter.

Acknowledgement – The authors gratefully acknowledge financial support provided by the National Science Foundation under Grant No: DMI-9908249. The zeta potential measurement carried out in the National Science Foundation Engineering Research Center for Particle Science and Technology at the University of Florida is also greatly appreciated.

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