Effect of dispersion parameters on the consolidation of starch-loaded hydroxyapatite slurry

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Abstract

The influence of some parameters controlling the hydroxyapatite (HA) suspension rheology in terms of heat treatment of the HA powder prior to suspension preparation, suspension solid loading and the amount of corn starch addition was thoroughly investigated. The heat treatment of powder at 1100 °C prior to suspension preparation was found to be extremely efficient in preparing suspensions with high solid loading of 59 vol.%. In contrast, the highest solid loading that could be developed from the non-heat treated powder was 14 vol.%. This phenomenon is consequence of the changes in the physical and chemical properties of the powder after the heat treatment step. The amount of native corn starch addition has ranged from 10 to 40 vol.%. The addition of corn starch leads to the high retardation in the suspension viscosity, particularly at low shear rate. On the contrary, at higher shear rate the situation is completely different. The properties of the consolidated green sample (produced from suspensions containing various corn starch amounts) in terms of relative density and compressive strength were studied. The results indicated that even though there were no considerable changes in the relative density, the compressive strength was sharply increased with increasing starch amount content until it reached a maximum at 30 vol.% and then decreased thereafter.

Keywords: hydroxyapatite, rheological properties, calcination temperatures, starch consolidation technique

I. Introduction

Calcium phosphate based bioceramics have received considerable attention as bone-graft substitutes, mainly because of their excellent biocompatibility, bioactivity and osteoconduction characteristics [1,2]. The most widely used calcium-phosphate-based bioceramics is hydroxyapatite (HA, $Ca_{10}(PO_4)_6(OH)_2$). Due to the chemical similarity between HA and mineralized bone of human tissue synthetic HA exhibits a strong affinity to the host hard tissues. The formation of chemical bond with the host tissue offers HA a greater advantage in clinical applications over most other bone substitutes, such as allograft or metallic implants [3]. However, the main drawback in the application of HA ceramics is its poor mechanical properties and the difficulties in fabricating parts with complex shapes [4]. Recently, a great effort has been made to overcome these problems via the trials in developing processing techniques able to achieve more reliable bioceramic bodies. The starch consolidation technique (which belongs to the category of direct consolidation technique) has been widely explored for shaping ceramic materials through the use of starch loaded in ceramic slurry [5,6]. This technique has been extensively applied for shaping ceramic powders, such as pure oxide ceramics [5,7–9], mullite [10,11] and cordierite ceramics [12,13], but for the hydroxyapatite systems it is still quite rare. The main advantage of using starch in ceramic forming, when compared to other biopolymers and natural polysaccharides, is in its chemical purity that secures residual-free burnout. Also, starch is easy to burn out and it is economically cheap and environmentally friendly for implementation in ceramic processing to produce ceramic materials with diverse applications [14].

However, preparation of high solid loading and well dispersed ceramic slurries is regarded as the main parameter deciding the power of practising this technique

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in the green ceramic body fabrication. The ceramic suspension should possess optimum rheological properties in order to be used in this technique. There are various parameters controlling the rheological properties of the ceramic suspensions such as: i) the amount and type of dispersing agent that should be added in order to deflocculate the suspension, ii) the properties of the ceramic powder (especially its physical properties in terms of particle size, surface area, pore volume, etc.), and iii) the type and amount of materials added during suspension preparation.

Some investigators [15,16] have mentioned that the properties of hydroxyapatite are largely influenced by heat treatment. The alterations in the properties of HA with heat treatment and the influence of these alterations in the behaviour of HA suspension are not thoroughly examined. Also, research of the effect of different starch amount addition on the rheological properties of ceramic suspension is rare. To the best of our knowledge, there are no reports dealing with the influence of starch amount on the rheology of hydroxyapatite suspension.

In this manuscript, the hydroxyapatite powder was firstly heat treated at 1100 °C and the influence of this treatment on the various physical and chemical properties of the powder was elucidated. The reflection of these changes (in powder properties via heat treatment) on the suspension rheology was studied. Also, a detailed rheological study is conducted for investigating the influence of hydroxyapatite solid loading on the suspension rheology. Additionally, the effect of adding various amounts of native corn starch on suspension rheology was investigated. Finally, the research on the effect of various corn starch amounts on the properties of consolidated green parts was carried out.

II. Experimental procedures

2.1. Materials

The hydroxyapatite powder used in this investigation was delivered by Riedel-de Haen Co., Seelze, Germany. The bulk characteristics, which are taken from the manufacturer, indicated that the powder only contains minor amounts of compounds other than calcium and phosphates (shown in Table 1). A stock of the powder was calcined at 1100 °C.

The dispersant applied in this investigation was Acumer 9400 supplied by The Dow Chemical Company, Midland, Michigan, USA. It is a water-soluble sodium salt polymer used to disperse and stabilize high-solid mineral slurries. It is an anionic dispersant of sodium polyacrylate polymer (SPA) with the solid content of 41–43 wt.% and molecular weight of 3000–4000 g/mol. The amount of SPA was fixed during the

Table 1. Impurities in as-received hydroxyapatite powders

Type of impurities	[%]
Chloride	0.15
Fluoride	0.005
Sulphate	0.5
Heavy metal	0.001
Fe	0.04
As	0.0002
Cd	0.0001
Cu	0.002
Zn	0.002
Loss of ignition (at 800 °C)	8

preparation of various HA suspensions (with and without corn starch) at 0.36 wt.% based on the total weight of hydroxyapatite. The temperature of heat treatment (1100 °C) and amount of dispersing agent (0.36 wt.%) were found to be optimal for preparing well dispersed HA suspensions according to the separate study currently submitted for publication elsewhere.

The commercial corn starch used in this work was supplied by El-Gomhoria Company, Egypt. This starch had no physical or chemical modifications. It was selected primarily due to its low price and easy reproducibility.

The physical properties of both hydroxyapatite powders (as-received and heat treated) and corn starch in terms of density and surface area are shown in Table 2.

2.2. Processing

Suspensions from both hydroxyapatite powders heat treated at 1100 °C and the as-received one were prepared by adding the predetermined amount of powder gradually to a predetermined amount of bi-distilled water under continuous stirring. The amount of Acumar required to stabilize the HA particles (0.36 wt.% based on the total weight of hydroxyapatite) was dissolved in bi-distilled water and then added to the suspensions. The pH of all suspensions was adjusted at 11. The solid loading of suspensions prepared from the heat treated HA powder varied from 14 to 59 vol.%. Afterwards, the suspensions were mixed in a planetary mill for 24 h using zirconia balls to avoid the agglomeration [17] and to allow the deflocculant to adsorb onto the particle surfaces. Volume ratio between suspension and balls was 2 in order to avoid grinding of the powder during mixing [18]. The HA suspensions (of 59 vol.%) were then transferred into a new pots and 10–40 vol.% of corn starch was added and mixed under vigorous contentious mechanical stirring for 3 h to ensure the homogenization of the suspensions. The total solid loading (HA and corn starch) of all suspensions was kept constant at 59 vol.%,

Table 2. Properties of as-received hydroxyapatite powder, calcined HA powder and corn starch

	As-received powder	Powder calcined at 1100 °C	Corn starch
Density [g/cm^3]	3.156	3.156	1.41
Surface area [m^2/g]	73.9	1.9	0.33

taking into consideration that the corn starch powder has lower density than hydroxyapatite powder (Table 2). The adjustment of the solid loading was achieved via the addition of predetermined amounts of distilled water during the addition of corn starch to the HA suspension. After 3 h of vigorous mechanical stirring for homogenization and conditioning the suspensions were then ready for casting. This adjustment of the solid loading was carried out in order to make the amount of added starch the only variable during rheological study and green sample properties determination. After casting the suspensions into non-porous moulds, they were heated in a dryer at 80 °C for 24 h. The drying process was carried out in an ordinary dryer oven Carbolite Fan Convection Oven (Hope Valley, UK) with a heating rate of 5 °C/min. According to the fact that the corn starch gelatinization started at 62 °C and ended at 80 °C [19], the drying temperature of all samples was fixed at 80 °C throughout the investigation.

2.3. Characterization

X-ray diffraction (XRD, Bruker axs D8, Karlsruhe, Germany) with CuK$_\alpha$ (λ = 1.5406 Å) radiation and secondary monochromator in the 2θ range from 20 to 100° was used to identify the formed phases and determine the crystallite size of the produced powder. The functional group analysis was performed by Fourier transform infrared spectroscopy (FT-IR). The measurements were carried out in the transmission mode in the mid-infrared range (400–4000 cm^{-1}) at the resolution of 4 cm^{-1}. The studies were performed using the instrument JASCO 3600, Tokyo, Japan. For FT-IR measurements, KBr pellets containing the exact weighted amount of the examined substance were prepared. Morphology of sintered samples was examined using scanning electron microscope (JEOL-JSM-5410 Tokyo, Japan) equipped with EDX unit (England, Oxford, ANCK). The particle size distribution of the powders was determined by the laser diffraction method (FRITSCH Model ANALY-SETTE 22, Idar-Oberstein, Germany). The specific surface area (S_{BET}) as well as the pore volume of the powders was determined by BET method using a surface area analyser (Autosorb-1, Quantachrome Instruments, USA). Zeta potential measurements were performed by a laser Zetameter (Malvern Instruments Model Zetasizer 200). A 0.1 g of sample was placed in 50 ml bi-distilled water with pH modifiers. The ionic strength of the medium was adjusted by adding 0.02 M NaCl solution. The suspension was conditioned for 30 h during which the pH was adjusted. After shaking, the equilibrium pH was recorded. 10 ml of the suspension was transferred into a standard cell for zeta potential measurement. Suspension temperature was maintained at 25 °C. Zeta potential was measured as a function of pH. 1M HNO3 and 1M NaOH were used to adjust the pH. Five measurements were taken and the average was reported as the measured zeta potential. The isoelectric point (*IEP*) was identified at the pH axis crossing point.

The rheology of the samples was determined using a Bohlin Rheometer (Bohlin Instruments Ltd, UK) with a cone-and-plate geometry. Experiments were performed at 25 °C. In all experiments, a cone with the radius of 60 mm and cone angle of 4.0° was used. Each sample was transferred to the rheometer plate at the desired temperature and excess material was wiped off with a spatula. The apparent viscosity versus shear rate curves were measured for HA suspensions with various volume fractions of the as-received and heat treated powder and for those containing various volume contents of starch. The bulk density and apparent porosity for both green and sintered samples were determined by Archimedes immersion technique. The compressive strength test was performed in order to evaluate the mechanical properties of the produced sintered samples. Samples of cylindrical shape (12 mm in diameter and 12 mm in length) were subjected to compressive tests. The test was performed at a crosshead speed of 0.5 mm/min using a universal testing machine. The compressive strength was evaluated from the maximum point of the stress/strain graph, which occurs when the first crack appears in the samples. The average of three samples was taken as the measure of the compressive strength of the sample.

III. Results and discussion

3.1. Rheology of HA suspension

Figure 1 illustrates the effect of both calcination temperature and HA solid loading on the rheological behaviour of HA suspensions. It is clear that all suspensions exhibit a shear thinning behaviour, whereas viscosity is largely decreases with increasing shear rate. The shear thinning behaviour is usually associated with the slurry structure. At low shear rate, liquid is immobilized in empty spaces within floc and floc network. As
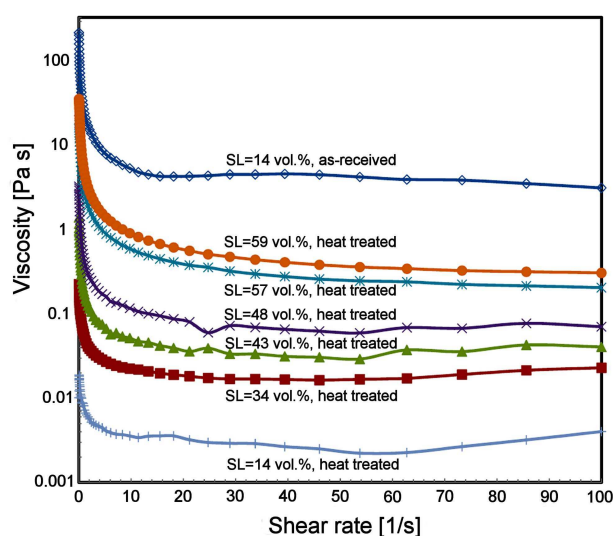
Figure 1. Viscosity-shear rate curves for suspensions prepared from as-received and heat treated hydroxyapatite powder containing different solid loading (*SL*)
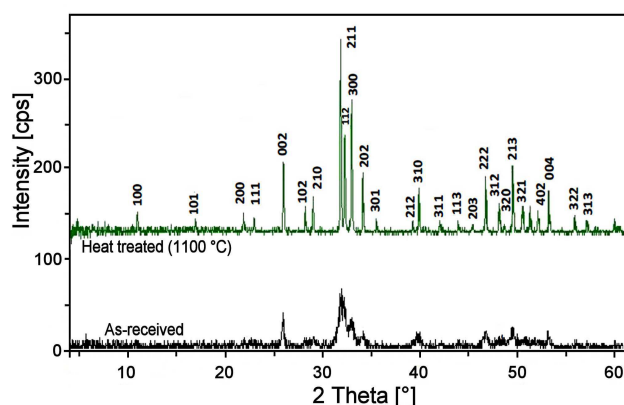
Figure 2. XRD patterns of both as-received hydroxyapatite powder and that heat treated at 1100 °C

Figure 3. FT-IR spectra of both as-received hydroxyapatite powder and that heat treated at 1100 °C

the shear rate increases, the floc and floc network break down, the entrapped liquid is released and a more ordered structure is formed in the flow direction [20].

However, 14 vol.% was found to be the maximum solid loading of suspension that could be produced from the as-received HA powder. In this case (for the as-received powder), when solid loading was above 14 vol.%, it was difficult to get dispersed suspension and actually the produced slurry had a paste like consistency. Accordingly, in the case of as-received HA powder the maximum solid loading could not exceed 14 vol.% due to more accentuated viscous character of this suspension. This situation is completely changed when the as-received powder is firstly calcined at 1100 °C before suspension preparation. Suspension containing the same solid loading (14 vol.%), but from the powder calcined at 1100 °C shows a sharp deterioration in the viscosity at the whole range of shear rate studied (Fig. 1). This is a reflection of the large enhancement in the flow behaviour of the suspension via heat treatment of the HA powder. The enhancement in the flow behaviour could be attributed to the changes in the chemical and physical properties of the HA powder that could occur after treating the powder at 1100 °C. Chemically, XRD patterns for the as-received powder and that calcined at 1100 °C (shown in Fig. 2) confirmed the presence of the same peaks related to hexagonal hydroxyapatite structure both in term of intensity and d-spacing compared with the standard HA in the powder diffraction file (PDF card # 9-432). No extra peaks reflecting the decomposition of the hydroxyapatite with heat treatment was identified. Although the as-received powder is composed of a pure hydroxyapatite it is clear from the pattern that the peak intensities of HA phase are very low for as-received sample compared to the same peaks for calcined ones. This observation could be owed to one or even both of the following reasons: i) the as-received sample is composed of a poorly crystalline HA with the presence of amorphous phase (with calcination of this sample at various temperatures the crystallinity degree of the HA phase is largely enhanced), ii) the particle size of the as-received sample is very
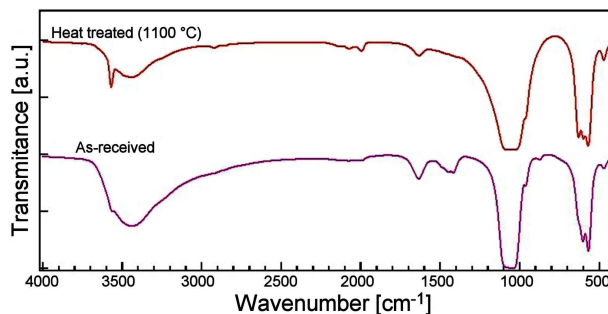
small. Upon calcination a grain growth of the HA particles is achieved forming large particles. This increase in sample particle size is responsible for increasing the peak intensities in the XRD patterns of the calcined samples. According to Scherrer's equation, the crystallite size of HA samples was increased from 28 nm for the as-received powder to 190 nm for powder calcined at 1100 °C. On the other hand, an overlapping of the 211 and 112 peaks at $2\theta \sim 32°$ is clearly noticeable for the as-received powder. This overlapping is an indication of the presence of hydroxyapatite phase in carbonated form [21]. The presence of carbonated HA in the as-received sample is confirmed by the FT-IR analysis of both samples. Figure 3, shows the FT-IR spectra for the as-received and calcined samples. In the spectrum of the as-received HA, beside bands characteristic for HA and water at $\sim$964, 470, 1045–1095, 570–605 cm^{-1} and 3444 and 1635 cm^{-1}, respectively, bands of CO_3^{2-} were clearly detected in the region around 1460, 1416 and 875 cm^{-1}. The band at $\sim$875 cm^{-1} indicates the v_2 mode of CO_3^{2-} group [22]. Moens *et al.* [23] and Xu *et al.* [24] have ascribed the appearance of bands at 872, 1417 and 1460 cm^{-1} to the presence of B-carbonated hydroxyapatite in which some of the CO_3^{2-} ions substitute the PO_4^{3-} ions in the hydroxyapatite structure. These bands are completely disappeared for the sample calcined at 1100 °C. The presence of this carbonate group has a reflection on the particle surface charge of the HA. The zeta potential measurements of two samples (the as-received and calcined, shown in Fig. 4) proved the shifting of the isoelectric point to more acidic region with heat treatment of the powder. This behaviour could be attributed to the presence of the carbonate group in the as-received powder. The presence of this group changes the surface behaviour of the powder making a shift in the isoelectric point to a higher basic region [25]. With heat treatment of the powder at 1100 °C this carbonate ions are almost eliminated (as confirmed from the FT-IR data shown in Fig. 3), which interpret the shifting in IEP of these powders to more acidic region. However, it could also be noticed that, with increasing the pH value, more negative charge is established on the surface of HA particles for both as-received and calcined samples. The zeta potentials were found to be negatively increased from ~ -3 for the as-received sample to ~ -29
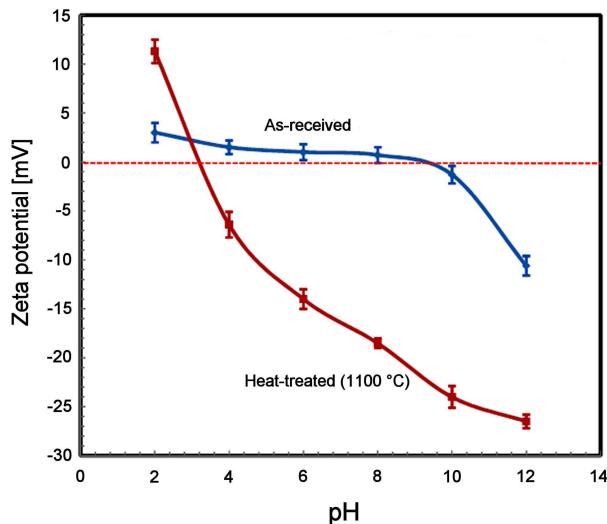
Figure 4. Zeta potential-pH curves of both as-received hydroxyapatite powder and that heat treated at 1100 °C

for sample heat treated at 1100 °C at pH = 11. This high negative charge on the surface of heat treated particles could be the reason for the enhancing in the flow behaviour of the suspension.

Physically, it was found that the surface area of the HA powder is largely reduced from about 74 m²/g for the as-received to ~2 m²/g for powder calcined at 1100 °C. The effective volume of liquid would be higher with utilization of lower surface area powder during suspension preparation. This means that higher solid loading suspension would be prepared from lower surface area powder. Moreover, it was found that the as-received powder is characterized by high pore volume fraction compared to that calcined at 1100 °C (the pore volume reduced from 0.119 cm³/g for the as-received powder to 0.002 cm³/g for the powder calcined at 1100 °C). The highly porous nature of the HA particles made each individual particle acts as a "sponge" absorbing liquid, and, thereby, increasing the effective solid loading in suspension [26,27]. In general, the heat treatment step highly affects both physical and chemical properties of the HA powder. This is the main reason responsible for the high decrease in viscosity of suspension prepared from the heat treated powder.

On the other hand, increasing the solid loading (14 to 59 vol.%) for HA powder calcined at 1100 °C leads to a sharp increase in the viscosity of the produced suspensions (as shown in Fig. 1). This behaviour was expected according to the fact that increasing solid loading leads to a decreasing in the amount of liquid in the system that could be present between particles and responsible for deflocculation [28]. However, the highest solid loading suspensions that could be produced from powder calcined at 1100 °C was found to be 59 vol.%. Beyond this value of solid loading the produced suspension had a paste like consistency. It is worth mentioning that the viscosity of the highest solid loading suspension produced from the heat treated HA (59 vol.%) is lower than that produce at much lower solid loading suspen-

sion from the as-received HA powder (14 vol.%). This indicates the effectiveness of the heat treatment step in changing the physico-chemical properties of the HA powder in order to prepare a high solid loading suspensions suitable for casting process. Moreover, the viscosity of all suspensions (with various solid loadings and except that produced from non-heat treated HA) is almost < 1 Pa·s at high shear rate = 100 s⁻¹ (Fig. 1). This indicates that all produced suspensions meet the requirements of the casting process [29].
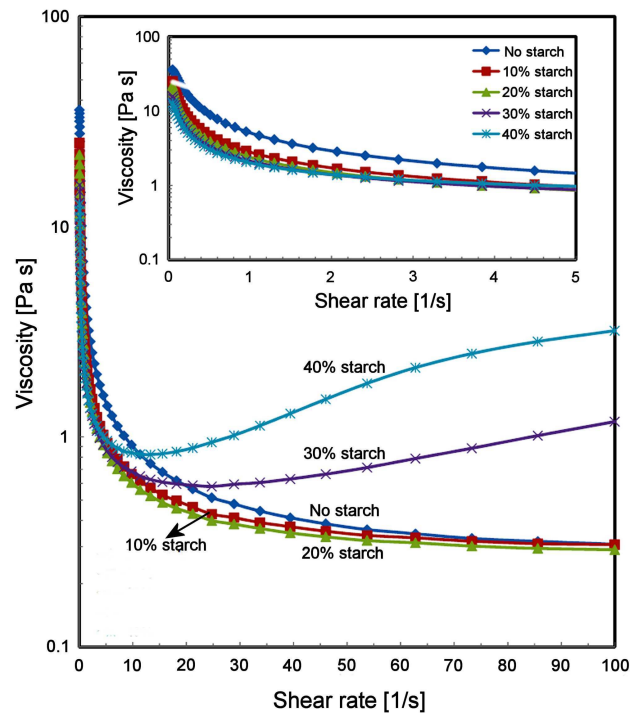
Figure 5. Viscosity-shear rate curves for suspensions prepared from heat treated hydroxyapatite powder containing different amount of corn starch

In order to investigate the effect of corn starch addition on the rheological properties of HA suspension, the suspensions from HA powder calcined at 1100 °C with various corn starch contents have been prepared. The corn starch content in the produced suspensions was varied from 0 to 40 vol.%, while the total solid loading was kept constant at (59 vol.%) which is the highest solid loading deduced from the previous study. Figure 5 shows the viscosity-shear rate curves for HA suspension prepared from HA calcined at 1100 °C and containing various corn starch content. There are some general features which are clearly noticeable. Firstly, increasing starch content leads to a considerable decrease in the viscosity, especially at low shear rate (shown as the insert in Fig. 5). Secondly, all suspensions with different corn starch content exhibit shear thinning flow behaviour. But, it is worthy to mention that, for suspensions with 10 and 20 vol.% starch exhibit this shear thinning behaviour at all ranges of applied shear rate, while the situation is different for suspensions containing 30 and 40 vol.% of starch. At 30 and 40 vol.% starch con-

tent, the suspensions exhibit shear thinning behaviour at low shear rate and then a shear thickening behaviour is pronounced at higher shear rate. The decrease in the viscosity of all suspensions with increasing starch content could be regarded as if starch weakens the inter-particle network in the HA suspension. All of the suspensions in the present study contained polyacrylate dispersant (0.36 wt.% based on the total weight of hydroxyapatite) which is known to stabilize the suspension with electrosteric means [30,31]. The weakening of inter-particle network with starch addition is believed to occur because of enhancing steric hindrance from the starch molecule (consisting of condensed glucose unit) that are possibly adsorbed onto the polyacrylate molecules [32]. Separate study showed that the pure starch suspensions (40 vol.%) display Newtonian behaviour [33]. On the other hand, in the present study it is confirmed that the pure HA suspension of ($\geq$40 vol.%) display shear thinning behaviour (deduced from the effect of HA solid loading on the suspension rheology). Herein, the solid loading of the final suspensions (with various starch additions) was kept constant. This means that the starch (whose pure suspension has near Newtonian behaviour) was added on the expense of HA powder (whose suspension has a shear thinning behaviour). This could be a convenient reason for the enhancing in the suspension fluidity with increasing starch amount added during HA suspension preparation.

The other obvious characteristic, deduced from Fig. 5, is the appearance of the shear thickening (dilatation) at high shear rates (the viscosity increases as the shear rate increases) for suspensions containing 30 and 40 vol.% of corn starch. Barnes [34] stated that shear thickening is a typical feature of well-deflocculated suspensions. The onset of shear thickening of the suspension is affected by solid loading, the particle size and shape, the polydispersity and the degree of stabilization. Hoffman [35] suggested that shear thickening is a consequence of an order-to-disorder transition. In well-stabilized suspensions, the particles are separated by a repulsive force and move individually, which leads to a low viscosity. The particles arrange themselves to be well separated; therefore, the structure is especially uniform (or "ordered"), compared to that of a flocculated suspension. Below the critical shear rate (the shear rate at which a transition from shear thinning to shear thickening occur), well-stabilized particles have a tendency to form a layered structure with close packing in the layers but higher separation between layers as a result of laminar flow [36]. At high shear rates, the layered arrangement becomes disrupted into a random three-dimensional structure [37]. This structural change requires greater packing space and presents an increase in the apparent viscosity. This could explain the reason for shear thickening behaviour of suspensions containing 30 and 40 vol.% of starch. Although the suspension solid loading is fixed at 59 vol.% the density of the corn starch is much lower compared to that of HA

powder. This means that the amount of fine powder introduced into the suspension increased with increasing corn starch addition. At higher corn starch amount (30 and 40 vol.%) the backing space required to stabilize the particles, particularly at higher shear rate, is diminished which is responsible for increasing the viscosity.

3.2. Properties of consolidated HA-corn starch suspensions

The relative density as well as compressive strength of the consolidated HA (0–40 vol.%) corn starch suspensions are shown in Fig. 6. The relative density was found to have insignificant changes with increasing corn starch amount. Whereas, the compressive strength of the dried sample is sharply increased with increasing corn starch addition until it reaches a maximum at 30 vol.% of corn starch and then it decreases thereafter. The unexpected constancy of the relative density with increased corn starch amount indicates that the addition of corn starch could not improve the packing ability of the producing mixture. This may be attributed to the fact that the surface area of the corn starch (0.333 m^2/g) was found to be much lower compared to that of HA powder (2 m^2/g). These results are in good agreement with Furnas' model [38]. According to his principle, the packing behaviour of a mixture should be improved when a portion of fine particles is replaced by the same amount of coarse one [39,40]. The improvement in sample compressive strength with additional starch amount is owed to the binding effect of the starch granules. The starch granules begin to swell by absorbing water at 55–60 °C during heating of the ceramic suspension, and then the starch granules are gelatinized gradually and the molecule chains extend above 60 °C. In fact, the swelling and gelatinization of the starch granules by absorbing water in situ reduce the free water in the ceramic suspension and the consolidation occurs, forming
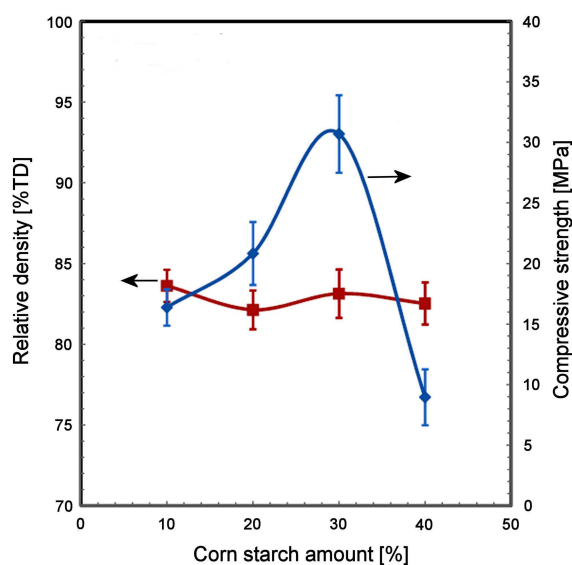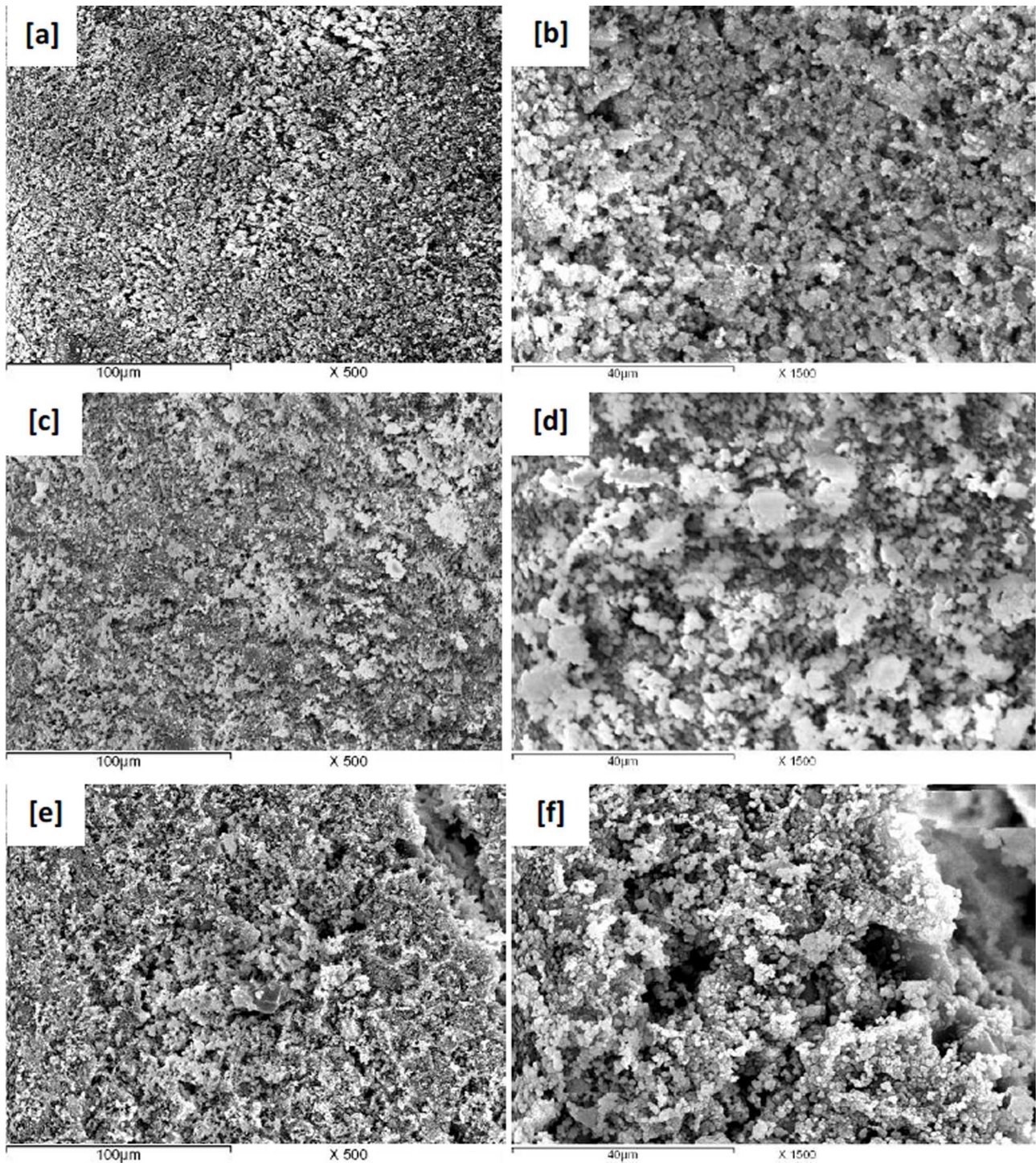

Figure 6. Effect of corn starch content on relative density and compressive strength of green HA samples

Figure 7. Microstructure of green HA sample containing different percentages of corn starch at two different magnifications: a,b) sample containing 10% corn starch, c,d) sample containing 30% corn starch and e,f) sample containing 40% corn starch

the green bodies. When the starch granules absorb water and swell, they create an extruding force among the ceramic particles, leading to the contact of ceramic particles. With the gelatinization of the starch granules, the molecule chains elongate in a great extent and permeate into the clearance among the hydroxyapatite particles and combine the particles. This promotes the consolidation of the ceramic suspension and increases the strength of the green bodies [41]. However, the decreasing of the compressive strength with increasing starch addition beyond 30 vol.% may be owed to the high shrinkage that would occur during drying. Lyckfeldt and Ferreira [33] studied the effect of starch addition on the density of the green ceramic sample and compared the experimental values with the predicted one for both high and low solid loading suspensions. They concluded that a good agreement between the experimental and predicted density values was found which means that there

are no obvious dimensional changes during the consolidation/drying process for suspension containing starch up to a specific content. Simultaneously, at high starch content there is some deviation between the experimental and predicted density values suggesting some shrinkage during drying.

The scanning electron micrographs of the samples containing 10, 30 and 40 vol.% of corn starch at both low and high magnifications are shown in Fig. 7. It can be seen that a homogeneous distribution of the corn starch was achieved in the final dried sample especially for sample containing low and medium starch amount (10 and 30 vol.%), Figs. 7a–d. However, with highest starch content of 40 vol.%, although there is a good starch distribution throughout the matrix a large crack was clearly noticeable with the presence of a large pore (Figs. 7e,f). This observation at the highest starch content can be related to a large quantity of shrinking starch granules during drying. A matrix with low density has a tendency to shrink during drying as there is, comparatively, much more empty space between hydroxyapatite particles. Furthermore, the shrinking of the starch can cause a local breakdown when adhered hydroxyapatite particles are removed, leaving more open space [33]. However, the presence of this crack in the sample containing 40 vol.% of corn starch easily explain the lower compressive strength of this sample.

IV. Conclusions

The heat treatment of the hydroxyapatite powder plays a highly versatile role in affecting the physicochemical properties of the powder and subsequently the rheological properties of the produced suspensions. A high solid loading of about 59 vol.% suspension could be prepared from HA powder heat treated at 1100 °C. In contrary, 14 vol.% was found to be the maximum solid loading suspension that could be prepared from the non-heat treated powder. Corn starch addition was found to be highly effective in enhancing the rheological properties of the prepared suspensions. The viscosity of high solid loading HA suspension is largely reduced with increasing corn starch addition from 10 to 40 vol.%, particularly at low shear rate. This makes the prepared suspensions with starch addition highly suitable for casting process. The green sample produced by a starch consolidation technique was found to be homogeneous with a uniform distribution of the starch particle in the HA matrix. The compressive strength of the green samples was found to be increased with increasing amount of starch up until 30 vol.% of addition, then it decreased thereafter. The decreasing in compressive strength beyond 30 vol.% of addition was owed to the high shrinkage of the starch, which causes a local breakdown when adhered hydroxyapatite particles are removed, leaving more open space.

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