ELSEVIER

Ceramics International 26 (2000) 279–287

CERAMICS INTERNATIONAL

Influence of dispersant on powders dispersion and properties of zirconia green compacts

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Received 8 March 1999; received in revised form 23 April 1999; accepted 10 May 1999

Abstract

Adsorption of water-soluble polymeric dispersant onto zirconia powders together with its influence on powder dispersion and green compact properties of the ceramics were investigated. Three different zirconia powders, each has its own specific particle morphology and surface chemistry (impurity), were employed. Typical Langmuir-type adsorption isotherms were observed for these zirconia powders; however, the isotherm plateau was roughly identical as approaching to maximum specific adsorption of approximately 2.5–2.8 mg per specific surface area of the powders, whilst the initial slopes in the isotherms were distinctly different which appeared to be powder-specific. This may arise from surface chemistry effect as further evidenced by an observation on the pyrolysis behavior of the adsorbance. The particle size and particle-size distribution of the powders generally became smaller and narrower, respectively, when the maximum adsorption was attained. Variation in particle size distribution gives rise to a significant effect on particle packing efficiency in the green-shape consolidates.

Keywords: A. Shaping; A. Suspensions; B. Microstructure-prefiring; B. Porosity; D. ZrO_2

1. Introduction

The use of dispersing agents to achieve a desired suspension rheology facilitating subsequent processing is of prime consideration in colloidal processing which is commonly regarded as a better way to produce high-quality, high-performance, and reliable ceramic products [1–3]. One important goal to be achieved in colloidal processing is to fully disperse the powder into finely-divided particles in a liquid medium to avoid the generation of agglomerates. The presence of agglomerates in ceramic powders is known to reduce particle packing eficiency and in consequence deteriorates the desired properties of the final ceramic parts by generating defects and/or developing undesired microstructure [4–7].

The degree of powder dispersion in a liquid medium is essentially dependent upon chemical (e.g. surface impurity and chemical nature) and physical (e.g. particle size/shape, surface energy) characteristics of the powder and the solid-dispersant interfacial adsorption behavior for a given specifically-selective dispersant, particularly for the cases of steric and electrosteric stabilization. Both the solid and the dispersant factors are essentially inter-related in a variety and complex manner [8–11]. For steric stabilization, as the focus of the present investigation, oxide particles (zirconia in this study) in an aqueous medium were stabilized by the adsorption of polymeric macromolecules. In principle, the solubility of the polymeric materials in liquid media determines to a significant extent the adsorption behavior of the solid. A ''good'' solvent for the polymers usually leads to a ''poor'' adsorption of the polymer onto the solid surface and vice verse. Since polymeric adsorption on the solid surface provides a layer of organic barrier surrounding the particles to prevent particles from further attraction in suspensions, flocculation can be avoided to a significant extent or even completely eliminated. Therefore, the selection of an appropriate ''solvent'' is relatively important in determining the resulting solid-polymer adsorption phenomenon and suspension stability in consequence. In this investigation, a water-soluble polymeric dispersant was employed, which has readily been found to be an effective dispersing agent for oxides

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0272-8842/00/$20.00
PII: S0272-8842(99)00054-1

in this lab, to disperse zirconia powders of different chemical and physical natures. The adsorption behavior of the dispersant on zirconia powders and its influence on powder dispersion and resulting green compact properties were reported.

2. Experimental procedure

Three commercial zirconia powders (Tosoh, Japan), designated as ZA, ZB, and ZC, having a mean particle size of 0.45, 0.32, and 0.1 μm, respectively, were employed. The chemical impurity of the powders and corresponding specific surface area were given in Table 1. For adsorption measurement, powder of identical surface area (based on the specific surface area from supplier) was used as primary controlled parameter. Accordingly, aqueous suspensions containing 2, 1.08, and 0.84 vol% for ZA, ZB, and ZC powders, respectively, and 0–145 mg (per ml water) of the water-soluble polymeric dispersant (in 50% aqueous form) were prepared. The suspensions were mixed in a polyethylene jar with a fixed amount of identical-sized alumina grinding balls for 24 h. The 24-h time period of mixing is believed to be long enough for the powders to reach equilibrium adsorption of the dispersant from the aqueous solution. The suspension was centrifuged. Part of the powders obtained were washed with deionized water for several times to ensure that no residual dispersant other than those adsorbed was appeared on the powder surface. The powders were dried in vacuum at 40°C until used. Adsorption experiment of the dispersant was conducted using thermal gravimetric analysis (with an accuracy of ± 0.1 mg). Since no attempt is tried to alter the surface properties of the powders, the powders employed in this study are thus in their as-received state. To avoid possible measurement error in determining adsorption ability of the powders, powder obtained under identical preparation condition as that of the suspensions but virtually free of dispersant was prepared. The obtained adsorption data is thus a result of the relative difference in TGA values between dispersant-treated and dispersant-free powders. The measurement difference in duplicate experiments of the powders was found to be within 2.6% of the mean specific adsorption.

The mean particle size and particle size distribution of the powders in the suspensions were determined using a particle size analyzer (Horiba, A-930). The green compact properties such as density and pore size distribution of the ceramics were measured using the mercury porosimetry (Autopore 9220).

Table 1
Impurity content, specific surface area (SSA), and average particle size (D_{ave}) of the ZrO_2 powders employed in this investigation

Powder	Impurity (wt%)		SSA (m^2/g)	D_{ave} (μm)
ZA	Y_2O_3	5.34%		
	CaO	0.02%		
	Na_2O	0.01%	6.8	0.45
	Ig. loss[a]	0.14%		
ZB	Y_2O_3	5%		
	CaO	0.02%		
	SiO_2	0.02%		
	Al_2O_3	0.02%	12.58	0.32
	Fe_2O_3	0.024%		
	TiO_2	0.046%		
ZC	Y_2O_3	5%		
	SiO_2	0.002%		
	Al_2O_3	0.005%	16.2	0.1
	Fe_2O_3	0.002%		
	Na_2O	0.025%		
	Ig. loss[a]	0.081%		

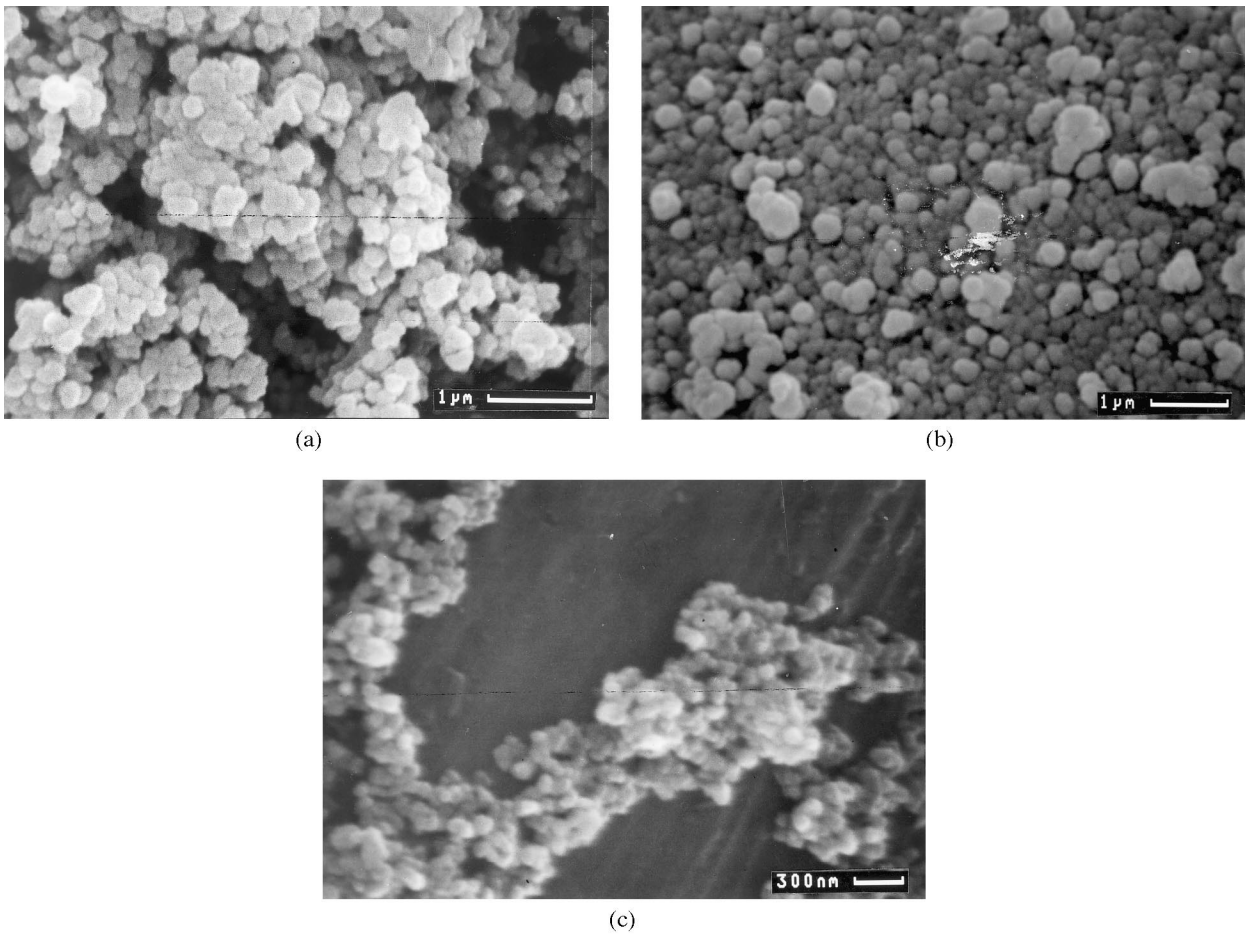
[a] Ig. loss = Ignition loss.

3. Results and discussion

3.1. Powder characteristics

Fig. 1(a)–(c) show the micrographs of the powder morphology for the ZA, ZB, and ZC powders, respectively. The actual mean size of the powders is greater than that from supplier and has an average value determined using scanning electron microscopy to be approximately 0.3, 0.22, and 0.08 μm for ZA, ZB, and ZC powders, respectively. The difference in particle size between the measured and supplier's value is believed to originate from powder agglomeration. The particles are essentially non-spherical in shape and some are present in the form of particle clusters, i.e. powder agglomerates.

Fig. 2 shows the influence of dispersant concentration on the mean particle size, i.e. d_{50}, of the powders. The d_{50} values for the powders made from dispersant-free suspensions is generally in the range of 1–1.5 μm, which is much greater than the values obtained from both the supplier and the SEM observation. This indicates an extensive powder agglomeration, particularly for the ZA powder which has the smallest mean particle size (having a nano-scale particle dimension) used in this investigation. The variation in d_{50} with dispersant concentration shows an increase in d_{50} at lower concentrations below about 50 mg/ml. Such an increase in d_{50} at lower dispersant concentration is believed to be a result of flocculation. The degree of flocculation, corresponding to an increase in d_{50}, in the suspensions appears to be powder-size dependent. The smaller of the particle, e.g. ZC powder, the greater degree of flocculation results and the most extensive flocculation in the suspension reflects in an increased valus of d_{50} by a factor of 4.5, 25, and 50% for ZA, ZB, and ZC powders, respectively. Such a powder flocculation in the suspensions at lower dispersant concentrations may be due to an incomplete adsorption of the dispersant on the particles.



(a) (b)

(c)

Fig. 1. Powder morphology for the (a) ZA, (b) ZB, and (c) ZC powders.

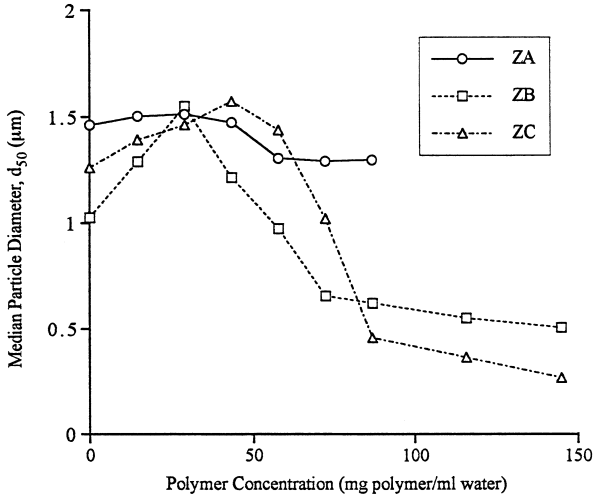


Fig. 2. The median particle size in the suspension changes as a function of dispersant concentration for different powders.

This deficiency may give rise to molecularly-level inhomogeneity on dispersant distribution at solid–polymer interfaces and a well-known phenomenon termed bridging (or necklacing) flocculation may provide a better understanding on such phenomenon.

Further increase in dispersant concentration effectively reduces d_{50} for powders ZB and ZC, suggesting agglomerates in these powders are essentially soft agglomerates (i.e. the attached particles were easy to separate from within the agglomerates under low to moderate shear field). Powder ZA, however, shows little change in d_{50} for dispersant concentration up to 145 mg/ml (not shown in Fig. 2). This indicates the powder agglomerates in ZA are primarily hard agglomerates (i.e. the particles were firmly attached with each other and a relatively higher shear field is usually required to separate them). The particle size distribution of the powders is becoming much narrower with increasing dispersant as selectively illustrated in Fig. 3 (a) and (b) for the ZB and ZC powders, respectively. It is interesting to note that the particle size distribution of the powders is likely to undergo a change from mono-modal to bi-modal, and then to mono-modal characteristic over the dispersant concentration range from approximately 58 to 87 mg/ml. This first transition in size distribution for the powders appears to lie nearby a region close to the onset of maximum adsorption as the dispersant concentration greater than 58 mg/ml for ZB powder and greater than 72.5 mg/ml for ZC powder.

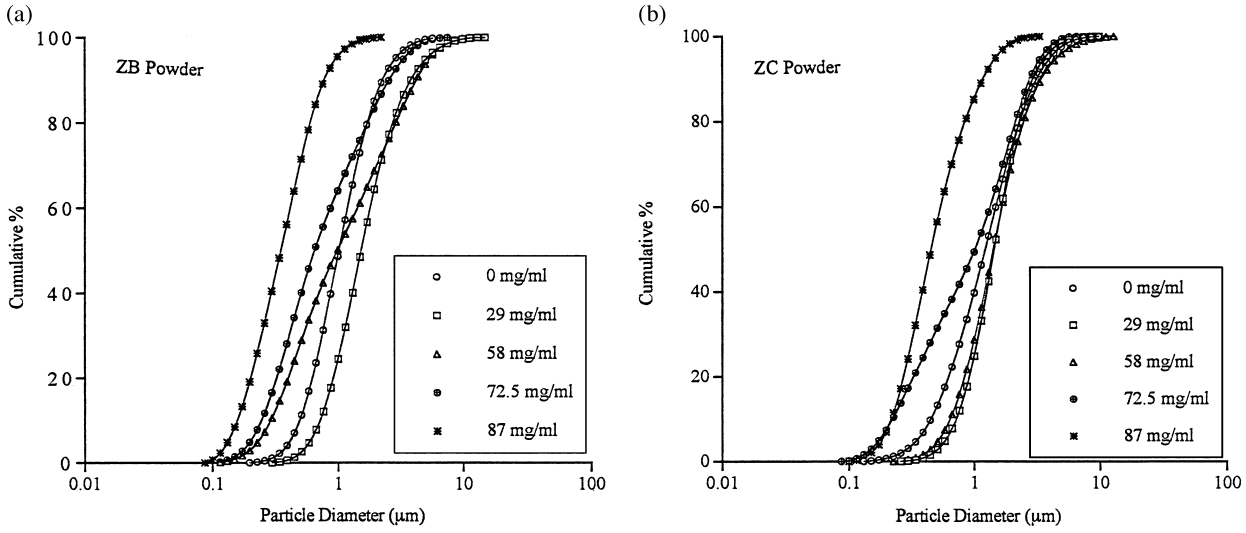


Fig. 3. The particle size distribution for the (a) ZB and (b) ZC powders at different dispersant concentrations.

This is due to the fact that the fractured agglomerates can be well-stabilized as that of the finely-divided particles in the suspension. The dispersant is sufficient to keep the suspension stable by effective solid-dispersant adsorption and a further increase in dispersant concentration permits a further size reduction of the residual agglomerates to some extent, i.e. the second transition.

The extensive reduction in mean particle (or agglomerate) size for powders ZB and ZC due to increased dispersant concentration may be accounted for by the fact that a reduction of particle size (d) accompanies an increase in specific surface area (SSA) of the powders according to,

$$d = 6/\rho.\mathrm{SSA} \quad (1)$$

where ρ is the solid density of the particles. Previous experimental observation strongly suggests that the newly-created surfaces effectively adsorbed the dispersant and allows a further steric stabilization of the suspension, provided that the concentration of dispersant is sufficiently enough to maintain a new equilibrium adsorption at the newly-formed solid–polymer interfaces. In other words, if the dispersant concentration is insufficient to effectively provide a complete adsorption at the interfaces, a further reduction in agglomerate size and stabilization of the suspension may probably not be achieved.

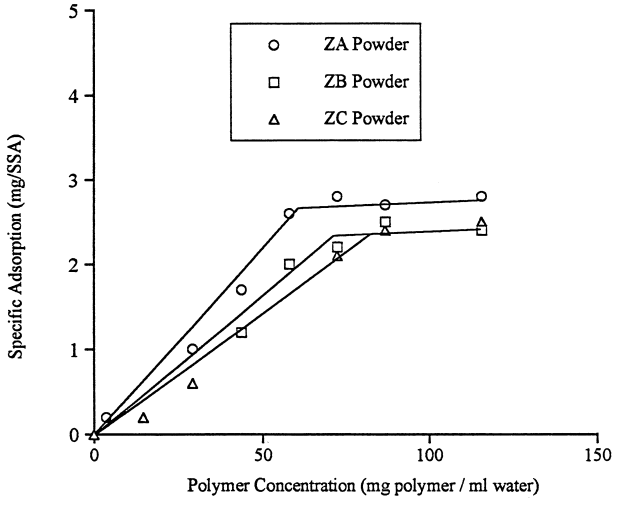


Fig. 4. The adsorption isotherms of three different powders.

3.2. Adsorption isotherm

The adsorption behavior of the dispersant onto the powders are expressed in terms of specific surface area (SSA) of the powders primarily because a variation in particle size (corresponding to a variation in SSA and will be discussed in later sections) was observed for these powder during adsorption experiment. Fig. 4 shows the resulting adsorption isotherms which exhibit a typical of Langmuir-type adsorption. Accordingly, such adsorption behavior suggests a monolayer adsorption of the dispersant onto the powder surface. An isotherm plateau corresponding to a complete monolayer adsorption reached at values of 2.5–2.8 mg polymer/SSA for these powders, where ZA powder appears to adsorb somewhat greater amount of the dispersant than the others. However, two interesting behaviors in the isotherms among the powders were observed. First, the dispersant concentration at which monolayer adsorption is reached appears to be powder-type dependent. The smaller the powder, the higher the dispersant concentration is required to reach maximum adsorption, i.e. ~50–58 mg/ml for ZA powder, ~72–75 mg/ml for ZB powder, and ~80–85 mg/ml for ZC powder. Secondly, the initial adsorption behavior (i.e. initial slopes in the isotherms) are distinctly different among the

powders, i.e. the larger the mean particle size, the greater the specific adsorption can be attained.

A large increase in newly-formed surface area due to extensive reduction in agglomerate size [according to Eq. (1) and Fig. 2] for powders ZB and ZC may provide some clues for the understanding of the first observation. Higher dispersant concentration is essentially required to provide effective adsorption of the dispersant on solid surface. For a given larger surface area, greater amount of dispersant is likely to be adsorbed and thus a higher dispersant concentration appears to be reasonably needed to stabilize the suspension. This can be further verified by a simple sediment experiment over which the stabilization of the suspensions can be detected as a function of time. For the case of ZC powder, about 4 weeks longer was required for the powder suspended at dispersant concentration of 87 mg/ml to settle than that a dispersant concentration of 58 mg/ml.

On reaching to the maximum adsorption, ZA powder has a specific adsorption greater by approximately 12% than that of the others. One possible explanation may be rationalized by assuming that the species adsorbed on the ZA particle surfaces in an orientation that permits a higher extent of adsorption than on the other powder surfaces. In such a way, the adsorption layer on ZA particle would be greater in thickness or more compact in adsorption density than on the other powders. Unfortunately, an attempt to calculate the thickness of adsorbed layer by means of Einstein's equation for very dilute suspension through a viscometric determination [12,13] failed primarily because of the presence of agglomerates (more or less for all the powders currently used) which may give rise to a considerable error in the estimate of effective solid loading in the suspension due to their tendency of entrapping liquid medium [14,15]. The "entrapped" liquid causes an increase in effective solid loading and this would usually result in overestimate of the adsorbed layer thickness. Difference in surface chemistry among these powders should play a crucial role in the adsorption as well as dispersion behavior [16,17]; however, the complexity of the surface chemistry–adsorption interaction makes a quantitative understanding of the adsorption behavior more difficult.

The adsorption results shown in Fig. 1 with a Langmuir adsorption model can accordingly be described by:

$$C = C_e K C_s/(1 + KC_s) \quad (2)$$

where C is adsorbed amount, C_e adsorbed amount at equilibrium (i.e. plateau region), C_s equilibrium concentration of supernatant, and K affinity constant. The slope of the initial straight line is equivalent to the affinity constant K. The higher value of the slope indicates a greater solid–dispersant affinity. In this case, the dispersant is likely to exhibit a decreasing affinity with respect to the solid surface in the order of ZA > ZB > ZC powder. This suggests that the powder has a surface characteristic more favorable to interact either chemically or physically with the dispersant molecules in an aqueous environment in the order of ZA > ZB > ZC.

3.3. Pyrolysis of adsorbent

Fig. 5 shows the resulting weight loss curves of the pure dispersant and the adsorbance (samples were collected from the powders equilibrated at the plateau region in the isotherm at a dispersant concentration of 72.5 mg/ml) for these powders. Obviously, the pyrolytic behavior of the adsorbent appears to be powder-type dependent. A higher temperature is usually needed to completely pyrolyze the adsorbent from ZA powder (510°C) than that from ZB (460°C) powder, and than ZC powder (400°C). This observation suggests a distinctly different routes for adsorbent "burnout" and the highest energy seems to be required for the adsorbent to "drive off" from the ZA surface than the others. A detail analysis of the mechanism would involve a surface catalytic effect together with the need for identification of possible decomposed species; however, it is beyond the scope of current purpose of investigation.

As a first approximation, we evaluate such powder-dependent pyrolysis from energy viewpoint. The energy required to pyrolyze the adsorbent from the surface may simply involve the energy for chain breakage (decomposition) and for evaporation. The energy for desorption of some relatively small decomposed species (if they exist) from the surface may probably be involved. As one realized, endothermic and exothermic peaks will both appear depending on the nature of reaction. The energy value of the test samples was determined by integrating the area under the DTA curve (temperature vs. relative change in microwatts or microvoltage) and

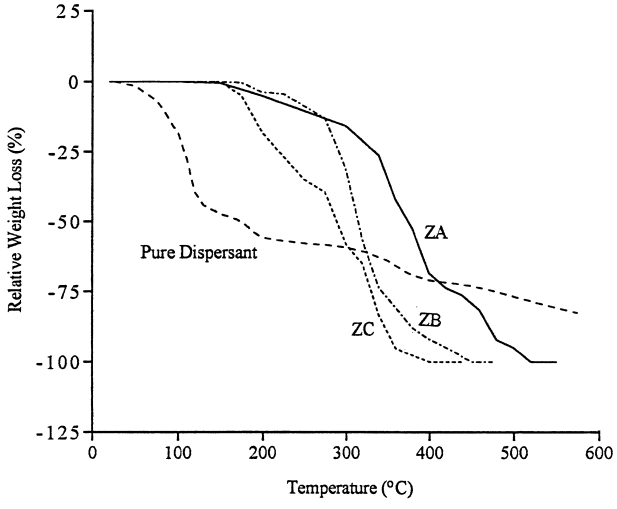


Fig. 5. The weight loss behavior for both the pure dispersant and the adsorbance on different powders.

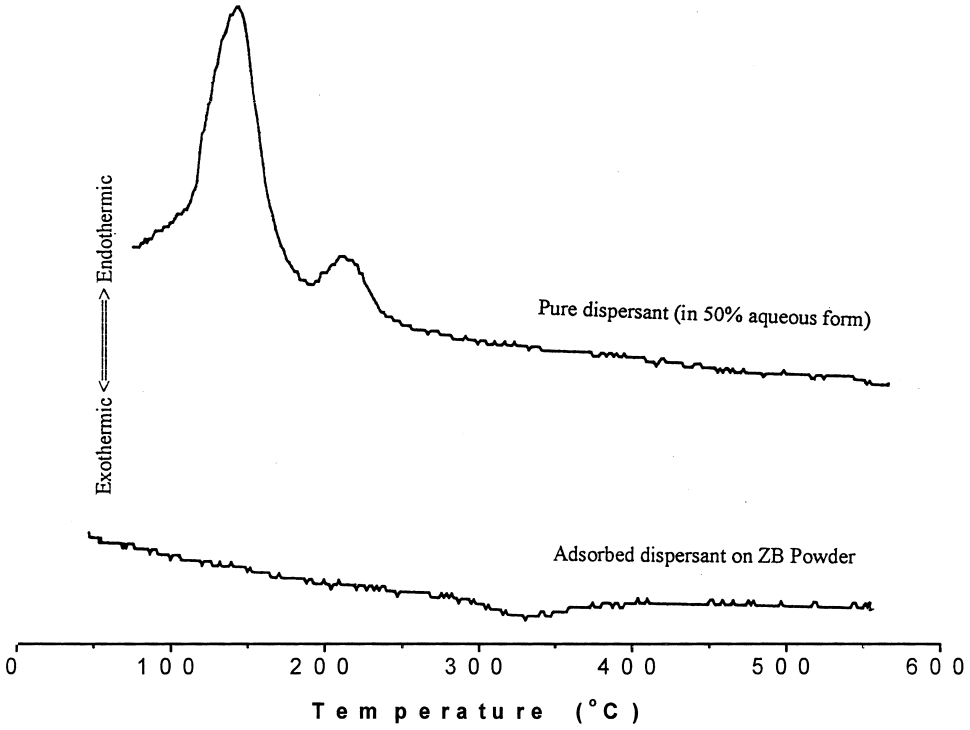


Fig. 6. The DTA spectra for the pure dispersant and the adsorbance on ZB powder.

calculating through the computer equipped with the instrument. Fig. 6 shows the DTA spectra for both the pure dispersant and the adsorbent on the ZB powder. The first major endothermic peak occurred at 120°C for pure dispersant is due primarily to water evaporation and the second one at 202°C is caused by adsorbent decomposition. The energy for pure dispersant to be pyrolyzed was determined to be +0.977 J/g (the "+" sign is referred to as endothermic character). While being adsorbed, only one relatively broad exothermic peak (characterized by "−" sign) was observed in the spectra suggesting the adsorbance undergoes a relatively slow pyrolytic reaction. The energy used to pyrolyze the organics for both samples revealed distinctly different characteristics, i.e. from endothermic characteristic for pure organic species to exothermic nature as the species being adsorbed. Since the primary difference between the test samples is the presence of a solid surface for the adsorbed dispersant, this forces us to realize in only one possible alternative, at least presently, that it is caused by surface catalytic effect. Such effect would largely alter the reaction nature of the polymer during pyrolysis when the polymer is adsorbed onto a specific solid surface, which in some aspects is closely resemble that observed in a binder burnout study of numerous oxide-polymer systems conducted by Masia et al. [18]. The markedly difference in the weight loss behavior in Fig. 5 between the pure dispersant and adsorbent may also provide some evidence of the surface effect.

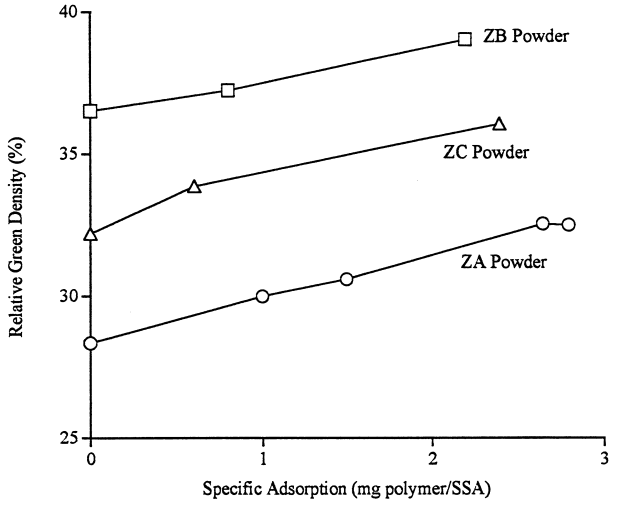


Fig. 7. The green compact density varies as a function of specific adsorption for different starting powders.

The energy required to pyrolyze the adsorbent from ZA powder is then determined to be −0.26 J/g, −0.183 J/g from ZB, and −0.11 J/g from ZC. The difference in energy values for adsorbent burnout from different powder surfaces suggests an effect primarily origimating from the variation in surface chemistry among the powders (Table 1). However, a poorly-defined surface characteristic of these powders can hardly make such a surface chemistry effect more clear in a quantitative manner. On a comparative basis, one may find that the energy for the pyrolysis of the adsorbent is in the same

(decreasing) order, i.e., ZA > ZB > ZC, as that of the solid–dispersant affinity previously discussed. Although no direct correlation can be quantified between these findings, it may be reasonably to assume that the "bonding strength" at zirconia–polymer interface may be related in some manner to the energy level required to pyrolyze the adsorbance.

3.4. Green compact property

The green density of the powder compacts obtained from centrifugal consolidation of the suspension is illustrated in Fig. 7 with respect to some corresponding specific adsorption values. Although the powders exhibit different packing densities for a corresponding specific adsorption, higher adsorption generally ensures a denser packing structure until a maximum adsorption is attained, e.g. for ZA powder. This finding appears to be coincident with the previous discussion on the mean particle size and particle-size distribution over the specific adsorption (Figs. 2 and 3). Higher adsorption tends to form more stable suspension with less powder agglomeration and thus result in a better particle packing eficiency as is evidenced in Fig. 7.

However, in certain cases particularly for suspensions with low dispersant, the pore distribution in the compacts shows essentially a bi-modal characteristic as illustrated in Fig. 8(a)–(c) for ZA, ZB, and ZC powders, respectively. These bi-modal pore distributions are believed to result from powder agglomerates. The agglomerates with a greater mass settle down in a higher rate than does the finely-divided particles under centrifugation. The agglomerates would therefore accumulate and construct as lower part of the consolidates. This separation between the particles and agglomerates would cause microstructural inhomogeneity and deteriorate the sintered property in consequence. If we defined the particle and the agglomerate as two separated phases, then the

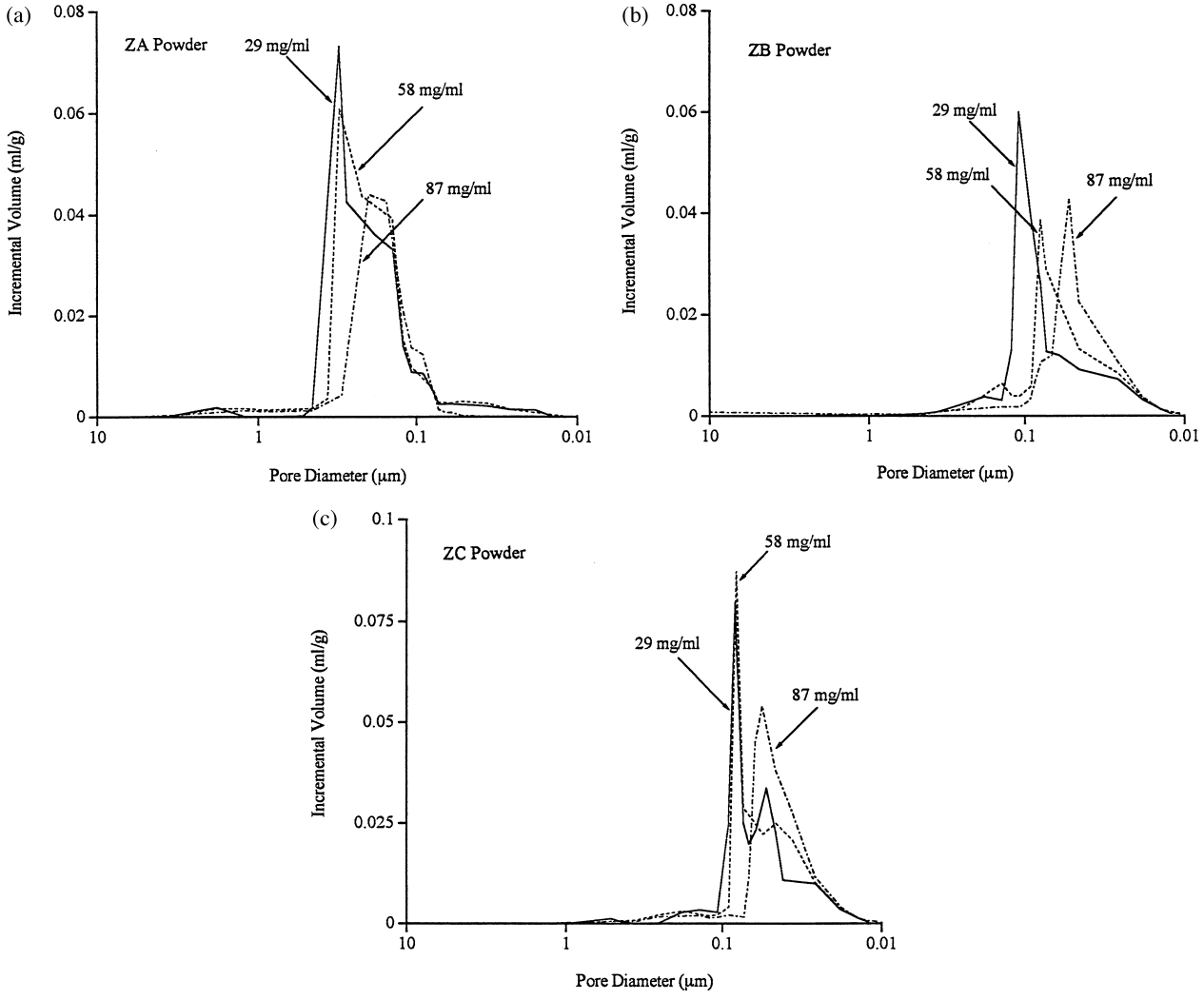


Fig. 8. The pore size distribution in the green compacts consolidated from (a) ZA, (b) ZB, and (c) ZC powder suspensions with dispersant concentration ranging from 29 to 87 mg/ml.

more separation of the phase, for instance, for powders with an extremely bi-modal particle size distribution, the greater degree of microstructural inhomogeneity would likely to be expected under current consolidation technique. This can be illustrated using the case of ZB powder. In ZB powder, a bi-modal particle size distribution is clearly differentiated (Fig. 3b) as in the suspension with dispersant concentration greater than about 58 mg/ml and the resulting pore distribution in the consolidate also reveals a bi-modal characteristic (Fig. 8b). However, at higher concentration, i.e. 87 mg/ml, both the pore and particle size distributions showed a nearly uni-modal (continuous) character.

Under centrifugating, the larger-mass agglomerates settled at a much higher rate than that of the finely-divided particles. The fast consolidation rate of the agglomerates makes no enough time for the agglomerates to rotate and/or move in a better packing orientation necessary to achieve a dense packing, resulting in a poor packing efficiency (Fig. 7) and the large voids may inevitably form. In the consolidates, the large pores, such as the pores over 1 μm for ZA powder, over 0.1 mm for ZB powder, and over 0.08 μm for ZC powder, are regarded to be caused by poor packing of the agglomerates. The smaller pores are believed to be constructed by packing of the finely-divided particles and by those of the intra-agglomerate voids. At higher dispersant concentrations (i.e. 87 mg/ml), the pore distribution tends to exhibit a uni-modal characteristic for particularly the ZB and ZC powders. The extensive size reduction in agglomerates as previously revealed in Fig. 2 for these powders at higher dispersant concentrations may be the major cause for this observation.

4. Conclusion

The adsorption of water-soluble polymeric dispersant onto zirconia powders and its influence on powder dispersion is investigated. The resulting green compact property of the consolidates after centrifugal consolidation is also characterized. The adsorption behavior of the powders exhibited a Langmuir-type adsorption isotherm over which a complete monolayer adsorption is likely to achieve at a maximum specific adsorption of 2.5–2.8 mg/SSA depending on the nature of the powder surface. A comparative examination of the pyrolysis behavior between the adsorbance and pure dispersant provides strong evidence of surface effect; however, it is rather difficult to analyze in a quantitative manner. At equilibrium, the degree of powder dispersion for the ZB and ZC powders is strongly related to the dispersant concentration in the solution, that is, the higher concentration of the dispersant, the greater degree of powder dispersion can be achieved and the suspensions turns to be more stable.

After consolidation, the green density as well as the pore distribution of the consolidates exhibited as a function directly related to the dispersing state of the powder in the suspension. In general, a higher adsorption of the dispersant results in a better particle packing efficiency and hence a higher green compact density. The size scale and distribution of the pores within the green consolidates are strongly related to the degree of powder agglomeration under centrifugal consolidation, e.g. powder with an increased inhomogeneity of particle size distribution tends to increase pore inhomogeneity in green compacts.

Acknowledgements

The author is gratefully indebted to the Minister of Economic Affair, ROC for funding toward this work under contract no. 863KG2230.

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