



Investigation of surface-modified anhydrous borax utilisation in raw glazes

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ABSTRACT

This study aims to determine the feasibility of preparing ceramic glaze using a surface-modified borate, which contributes boron to the composition without the need of a fritting process. In this context, surface modification of anhydrous borax powders (ABP) with magnesium stearate (MgSt) via dry powder coating is investigated. The surface modification of ABP with MgSt is optimised by employing modifier dosage of 0.5, 1, and 2 wt% and coating periods of 30, 60, and 120 min. The resulting powders are comparatively characterised via wettability, solubility, and dispersibility tests. The structural changes in surface-treated ABP are investigated using X-ray photoelectron spectroscopy (XPS) and Fourier-transform infrared spectroscopy (FT-IR) analyses. The results indicate that ABP surface could be switched from hydrophilic (17°) to hydrophobic (115°), its water solubility decreased from 40% to 10%, and a coating yield of approximately 74% was achieved with MgSt dosage of 1 wt% at a processing period of 2 h. Furthermore, FT-IR and XPS results indicated that MgSt is mainly coated over the surface of ABP via physical adsorption rather than chemical bonding. The glaze containing surface-treated ABP that was fired at 1050 °C, demonstrated complete melting and surface coverage without defects. Thus, effective dry coating as a single-step approach could be applied to obtain surface-modified ABP, which offers controlled solubility in glaze suspensions with improved dispersibility and excellent glaze coverage of the surfaces.

1. Introduction

Boron minerals are widely utilised in numerous industrial areas such as ceramic, glass, metallurgy, agriculture, medicine, cosmetics, automotive, communication, insulation, and energy [1]. Utilisation of boron in glass and ceramics industry improves the yields and reduces the energy requirement owing to its excellent fluxing and glass-forming properties [2].

The water-solubility of borates is a desirable property in some applications such as perborate-containing detergents, boronated fertilizers, additives for corrosion inhibitors, cutting fluids, insecticides, cosmetic, and pharmaceutical formulations [3]. It is known that most of the borates are partially water-soluble. Anhydrous borax ($\text{Na}_2\text{B}_4\text{O}_7$), for example, has a water-solubility value of 3.37 wt% at 25 °C [4]. However, it causes significant problems when used directly in ceramic glaze applications. The water in glaze suspension wicks into the interior of the body during application. During the drying process, the soluble material migrates along with water through the ridges or high areas of ceramic body, causing defects such as blistering and dry glaze areas [5]. Moreover, when used in glaze suspensions, the soluble borates increase the ionic strength and negatively affect the rheology. In order to prevent the above-mentioned application failures, a coating method that

can prevent the interaction of water with borax particles should be investigated.

Powder coating method focuses on improving the functionalities that are not inherent, and has been utilised for producing special chemicals and ceramics [7–10] under wet or dry conditions, depending on the limitations of the material to be coated [6]. In the dry method, host particles (1–500 µm) can be coated mechanically with guest particles (0.1–50 µm) in order to improve wettability, solubility, and other characteristics [6,11–13]. As the guest particles are too fine, the van der Waals interactions are sufficiently strong to keep them firmly attached to the host particles. Thus, either a discrete or continuous coating of guest particles can be achieved depending on the choice of equipment and operating conditions such as processing period, mechanical action, weight fraction of guest to host particles, and physical properties of the particles used [11,13,14].

Some of the dry coating applications in different domains are TiO_2 coating of poly(methyl methacrylate) particles using mechanofusion to improve the flowability, and magnesium stearate (MgSt) coating of silica gel using hybridiser and cyclomix to modify the wettability [6]. A planetary ball mill was used as an alternative promising tool by Sonoda et al. [15] to coat starch particles with a poorly water-soluble drug-flurbiprofen. Moreover, various organic [16–19] and inorganic powders

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[13,20,21] were selected as host particles in dry coating studies where magnesium stearate was used as the guest particle, and a surface coating with low wettability was achieved even if MgSt was used in negligible amounts.

Nowadays, a high-cost fritting process is applied as a technological precaution to render the boron compounds insoluble in water. This study aimed to apply a simple one-step method to demonstrate the effect of mechanical dry coating on the solubility of anhydrous borax powders (ABP) treated with magnesium stearate by using a planetary ball mill. The effects of the weight ratio of the guest/host powders and processing period on the surface properties of ABP were investigated. The powders with low-water solubility thus obtained were used in raw glaze to evaluate the effect of MgSt coating on ABP.

2. Experimental studies

2.1. Materials

ABP of purity 99% and size less than 0.5 mm was supplied by Eti Mine Enterprises (Turkey). ABP host particles were treated with 0.5, 1, and 2 wt% MgSt guest particles to yield different extents of surface coverage in dry coating processes. All the above percentage values are given as ratios in weight. The main properties of the guest particles are presented in Table 1.

2.2. Dry powder coating processes

As a one-step easy route, a dry process is preferred for simultaneously grinding, mixing, and coating powders by using a planetary ball mill, which consists of a ceramic jar (volume 500 mL) filled with alumina balls (diameter 10–20 mm). The rotational speed of the jar is 350 rpm, and a ball/powder weight ratio of 2:1 is used. The jar also contains a free volume of approximately 60% for the movement of powders.

The suggested dry coating process of ABP with MgSt in a planetary ball mill system can be summarised as follows. Based on our experiences, we believe that it is appropriate to mix ABP (100 g) with MgSt at mass fractions of 0.5%, 1%, and 2%. The processing periods were selected as 30, 60, and 120 min. After being processed, the powders were removed from the grinding jar and stored in a desiccator for subsequent characterisations.

2.3. Characterisation of surface-treated anhydrous borax powders

In order to determine the effectiveness of the applied process, the wettability, solubility, and dispersibility parameters were investigated. The effect of variables of the dry coating process on the particle size of ABP was also revealed.

2.3.1. Particle size distributions and solubilities of powders

The process kinetics was first analysed through variations in the size of powders caused by the variation in the processing period. The particle size distributions of as-received and surface-treated ABP were measured using a laser scattering particle analyser (Mastersizer 2000, Malvern Instruments Ltd., Worcestershire, UK). Three runs were performed for each sample, and the standard deviations were defined using

Table 1
Technical data of magnesium stearate.

Formula	$[\text{CH}_2(\text{CH}_2)_{16}\text{COO}]_2\text{Mg}$
Magnesium content (%)	4.6 ± 0.2
Magnesium oxide content (%)	6.5 ± 0.5
Free fatty acid (%)	less than 0.5
Melting point (°C)	200
Bulk density (gr/cm^3)	1.028
Particle size (μm)	$d_{10} = 0.15$; $d_{50} = 2.28$; $d_{90} = 5.82$

the “Compare” segment of the software.

2.3.2. Determination of the wettability of powders

The sessile drop method (KSV Attension ThetaLite TL 101 Optical Tensiometer), which involves depositing a small drop (five drops per sample) of pure water on tablets prepared using a uniaxial hydraulic press, was used for determining the wettability of the powders at 25 °C, in terms of the contact angle. The contact angle measurements were performed three times for each sample, and the average values were obtained.

2.3.3. Evaluation of the coating yield

As a water-soluble material, ABP has a strong tendency towards hydration; therefore, insoluble MgSt was employed to mitigate the water interaction of ABP. There are two main parameters affecting the surface hydration rate of ABP: increased surface area and the effectiveness of surface coating as indicated by the contact angle measurements. The two necessary requirements for obtaining serviceable powders are fine particle sizes and low water interaction and the combined effect of these factors is evaluated in terms of the obtained coating yield. After an extensive review, it was concluded that coating characterisation for soluble and non-soluble materials must differ. In the existing literature, coating was evaluated using an activation index [22,23], which is the weight ratio of the floating part on water to that of the overall sample for non-soluble materials. As the mass of soluble materials differs during the activation index test, a new method of assessment of effectiveness of coating is necessary. Thus, this study developed the index **coating yield**, which is the mass ratio between the ABP amount prevented from dissolving by surface treatment to the amount of dissolved ABP without surface treatment under the given conditions.

Coating yield assessment involved solubility tests, in which a maximum amount of soluble powder, represented by m_0 , was dissolved in 100 mL of water in a beaker of volume 250 mL, followed by magnetic stirring at 750 rpm and room temperature for 1 h. The insoluble parts of the powders were collected using a filter paper of pore size 4–12 μm and subsequently dried at 60 °C for 48–72 h and weighed sequentially ($m_{1,2}$) until a constant weight was reached. The drying temperature (60 °C) was chosen to be lower than the degradation temperature of MgSt. The amounts of water-insoluble powders of surface-treated (m_1) and untreated ABP (m_2) were measured for each processing period, and coating yield (%) was calculated according to Eq. (1). All solubility tests were performed three times and the values of m_0 , m_1 , and m_2 used in Eq. (1) were the calculated averages of these measurements.

$$\text{Coating yield (\%)} = [(m_1 - m_2)/m_0] \times 100 \quad (1)$$

2.3.4. Fourier-transform infrared spectroscopy (FT-IR) and X-ray photoelectron spectroscopy (XPS) analysis

The Perkin-Elmer Spectrum BX spectrophotometer equipped with an attenuated total reflectance attachment (ATR-FTIR) was used to detect the structural changes. The samples were scanned from 4000 to 500 cm^{-1} with a resolution of 4 cm^{-1} and the average of 25 repetitions per sample was obtained.

The surface chemistry of the samples was analysed using XPS (Thermo Scientific) with a monochromatic Al-K α (1486.7 eV) X-ray source and a spot size of diameter 400 μm . The device was calibrated according to gold (4f7/2). The vacuum pressure was less than 10^{-9} Torr during the spectral data acquisition. Survey XPS data were obtained in the range –10 to 1350 eV with the pass energy of 150 eV and resolution of 1 eV. Twenty scans were recorded from a single point. The pass energy for high-resolution elemental scanning was 30 eV with the scan number of 15. Binding energies, atomic concentration ratios, and deconvoluted spectra were obtained using a curve fitting software program.

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