Influent of Borax Decahydrate Composition as Additional Flux into Stoneware Bodies

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Abstract: Stoneware is vitrified, has less porosity and requires high sintering temperature. The influent of borax decahydrate composition at sintering temperature 1050°C and 1150°C on the thermal analysis, fracture surface, linear shrinkage, water absorption and modular of rapture (MOR) were investigated. Rectangular sample were produced by uniaxially pressing at 40MPa. The thermal behavior was determined by thermogravimetric and different thermal analysis (TGA-DTA). The Scanning electron microscopy (SEM) was used for fracture surface analysis. The water absorption (%) of the sample were determined using Archimedes' method. The experimental result showed that content of borax decahydrate have influent the properties of stoneware bodies.

1. Introduction

In general, definition of traditional ceramics is obtained from the range of product formed of bodies based on clay, fluxing or fluxed which is feldspar and filler such as alumina or quartz such as daily product of pottery and porcelain which refer to whiteware, refractory, cement and bottle glass. The common classes of traditional ceramics are as shown in Figure 1.1 [1][2]. In whiteware product, there a few materials such as porcelain, earthenware, dental porcelain, and stoneware.

Stoneware bodies does not need sintering to white product due to impurity content increase coming from secondary clay such as ball clay and has special characteristics [3][4]. The triaxial stoneware is formulated from a mixture of kaolinite clay (Al₂Si₂O₅(OH)₄), quartz (SiO₂) and flux [(K, Na, Ca) AlSi₃O₈]. The raw material composition for triaxial stoneware is typically within the range 40-60%wt clay, 20-35wt% flux and 20-30%wt quartz. If the ratio of raw material is changed or replaced by other material, the characteristics of stoneware will change [5][6][7].

In stoneware bodies, feldspar is the main source of flux. Low temperature of flux borax decahydrate (750°C-1000°C) which is below the temperature range of feldspar (1140-1150°C) will provided lower energy compared to high temperature process [9][10]. In the present work, influent of borax decahydrate content as as additional flux was examined for stoneware bodies. The sintered specimens were characterized and the obtained results were discussed.
2. Experimental procedures
In this study, sample was prepared by using stoneware bodies. The raw material from stoneware bodies were dried in an oven (PF20,Carbolite) 110°C for 24 hours subsequently crush by using Los Angeles Abrasion machine with speed 800rpm and sieve through less than 80µm. For borax decahydrate, the particle size was in range 80µm. Subsequently, the raw material from stoneware bodies also 3wt%, 4wt%, 5wt%, 7wt% and 10wt% of borax decahydrate (BD) respectively were added into the system. All the composition were dry milled using zirconia ball milled for 30 minute with speed 250rpm in order to homogenize the sample. To produce 8gram rectangular sample with 5.5mm height, sample was pressed in rectangular die-pressing of 65mm x 12mm under pressure 40MPa with holding time 1 min using Carver Bench Top Standard Auto Series. Rectangular sample were sinter at temperatures (1050°C and 1150°C) in electrical furnace (NABERTHERM) at the rate 5°C/min, with soaking time 2 hour respectively. After sintering, sample was cooled naturally to room temperature.

2.1 Characterization of sintered sample
Crystalline phases from stoneware bodies and borax decahydrate was performed using x-ray diffraction (XRD - Rigaku ultima IV). The thermal behavior raw material from stoneware industry and fluxes was determined by thermogravimetric and differential thermal analysis TGA–DTA (Rigaku-tg 8120 thermo plus). The fracture surface of the sintered samples was observed by scanning electron microscopy, SEM (Hitachi SU1510) and etching by using HF4% for 3 second.

2.2 Physical and mechanical properties
Properties such as water absorption (%) was determined according to standard procedure ASTM C373. Weight gain of dried sample after immersion into boiling water for 5 hours, soaking for 24 hours and seeping the surface with wet towel. For the modular of rapture (MOR), rectangular is suspended between two points, a force is applied in the center and the elongation of the sample is measured (speed 1mm/min) using Universal Testing Machine (GOTECH AI-7000S) based on ASTM C674.

3. Results and discussion
3.1 Crystalline phases of stoneware bodies and borax decahydrate
The XRD pattern of stoneware bodies and borax decahydrate are shown in Figure 3.1 (a) and Figure 3.1 (b). The major crystalline phases in stoneware bodies are kaolinite (JCPDS NO. 006-0221), illite (JCPDS NO. 043-0685), quartz (JCPDS NO.046-1045) and microline (JCPDS NO.001-0705). Meanwhile, borax decahydrate consist borax (JCPDS NO. 024-1055), Tincaleonite (JCPDS NO. 007-0277) and Calcite magnesian (JCPDS NO. 043-0697).
3.2 TGA-DTA result
As can be seen from Figure 3.2 (a), the weight loss (mg) of stoneware bodies with additional BD10wt% and BD5wt% gradually increases than stoneware bodies. The percentage weight loss (mg) of stoneware bodies with addition of BD10wt% is 4.467% and BD5wt% is 4.430wt%, which is the highest in stoneware bodies 4.000%. Figure 3.2 (b) shows that industrial stoneware bodies with additional BD5wt% and BD10wt% provide viscous liquid at temperature 1044.203°C and 1103.584°C lower than stoneware bodies.

3.3 Fracture surface of sample
Now turning to fracture surface of industrial stoneware bodies with BD10wt% sintered at temperature 1050°C in Figure 3.3 (a). When the sample was sintered at temperature similar with stoneware bodies, it can be seen from the both small pores compared stoneware bodies with BD10wt% sinter at temperature 1150°C.

On the other hand, stoneware bodies with BD10wt% sintered at temperature similar to temperature of stoneware bodies not only increase the closed pores but at the same time deteriorate the surface of the sample as shown in Figure 3.4. The most obvious finding to emerge from Figure 3.4 is clear cracks and open pores can be seen. In addition, pores were regarded as flaw which could be a predominant factor of deteriorating the strength.
Figure 3.3: (a) Fracture surface of stoneware bodies with BD10wt% sinter at temperature 1050°C and (b) Fracture surface of stoneware bodies sinter at temperature 1150°C.

Figure 3.4: Fracture surface of stoneware bodies with BD10wt% sinter at temperature 1150°C.

3.4 Linear shrinkage
The value of linear shrinkage shows an overall steady increase with BD3wt%, BD4wt%, BD5wt%, and BD7wt% into industrial stoneware bodies at temperature 1050°C in Figure 3.4. It clearly shows industrial stoneware bodies with BD10wt% 7.910 ± 0.206 % shrinkage faster at temperature 1050°C compared to stoneware bodies sintered at 1150°C that contracts at 10.210 ± 0.721 %. Linear shrinkage is reduction of length of sample during sintering and standard properties of linear shrinkage of stoneware bodies usually between 10% to 15% (Landberger & Lundin, 2013), thus the value of linear shrinkage obtained does not engage with the requirement. A viscous flow of liquid phase during sintering strongly effects the percentage of linear shrinkage.

3.5 Water absorption
Figure 3.5 reveals that there has been a sharp decrease of stoneware bodies with BD10wt% at temperature 1050°C attributed to water absorption which is directly correlated to complete viscous flow of liquid phase provided by fluxed. In fact, water absorption decreases with increase of sintering temperature due to formation of liquid phase and low value of water absorption indicating high degree of vitrification.

Clearly from the findings that industrial stoneware bodies with BD10wt% sintered at temperature 1050°C give the lowest value of water absorption 0.449 ± 1.210. Perhaps the most important compelling, the value obtained in stoneware bodies with BD10wt% at temperature 1050°C is fewer than 82.245% compared to stoneware bodies 2.529% sintered at temperature 1150°C. Increasing of water absorption
of stoneware bodies with BD10wt% at temperature 1150°C results from effects of growth of closed pores as revealed in [5].

![Figure 3.5](image)

**Figure 3.5:** The influence of borax decahydrate on (a) linear shrinkage (b) water absorption.

### 3.6 Modular of Rapture

Figure 3.6 displays the experimental data of content borax decahydrate at different weight percent. Figure 3.6 illustrates that with BD10wt% of borax decahydrate the modular of rapture increase from 23.580 ± 0.916 MPa to about 51.446 ± 3.607 MPa. However, industrial stoneware bodies containing BD3wt%, BD4wt%, BD5wt% and BD7wt% are sintered at 1050°C were about 41.937 ± 2.140 MPa. It can be seen that the value is higher when compared to stoneware bodies sinter at temperature 1050°C 20.553 ± 1.990 MPa.

Thus the presence of other important fluxing agents such as boron (B) could lower the sintering temperature and improve mechanical properties. Meanwhile, when all the samples containing borax decahydrate are sintered at temperature 1150°C, it will deteriorate the surface of structure as seen in Figure 3.4 and decrease the strength of sample.

![Figure 3.6](image)

**Figure 3.6:** The influence of borax decahydrate on modular of rapture (MOR).

### 4. Summary

The influence of borax decahydrate composition as additional flux were studied. Borax decahydrate can replace conventional flux such as sodium or potassium feldspar in stoneware bodies. In summary optimum borax decahydrate which is 10wt% basically lower the water absorption and increase the modular of rapture.

### References

[1] Cardarelli, F. (2008). Materials Handbook: A Concise Desktop Reference: Springer London.
[2] Somiya, S. (2012). *Advanced Technical Ceramics*: Elsevier Science.
[3] Boch, P., & Nièpce, J. C. (2010). Ceramic Materials: Processes, Properties, and Applications: Wiley.

[4] Singer, F. (2013). Industrial Ceramics: Springer Netherlands.

[5] Das, S. K., & Dana, K. (2003). Differences in densification behaviour of K- and Na-feldspar containing porcelain bodies Thermochimica Acta (Vol. 406, pp. 199-206).

[6] Buchanan, R. C. (2004). Ceramic Materials for Electronics, Third Edition: Taylor & Francis.

[7] Chakraborty, A. K. (2013). Phase Transformation of Kaolinite Clay: Springer India.

[8] Sharma, P. C. (2007). Production Technology (Manufacturing Processes): Manufacturing Processes: S. Chand.

[9] Gennaro, R., Cappelletti, P., Cerri, G., de’ Gennaro, M., Dondi, M., Guarini, G., . . . Naimo, D. (2003). Influence of zeolites on the sintering and technological properties of porcelain stoneware tiles Journal of the European Ceramic Society (Vol. 23, pp. 2237-2245).

[10] Mukherjee, S. (2012). Applied Mineralogy: Applications in Industry and Environment: Springer Netherlands.