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PREPARATION, CHARACTERIZATION, AND THERMAL STABILITY OF B_2O_3-SiO_2

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ABSTRACT

Preparation of B_2O_3-SiO_2 compound by inorganic synthesis was carried out. B_2O_3-SiO_2 was characterized by FT-IR spectrophotometer, analysis of crystallinity by XRD, and test of acidity. B_2O_3-SiO_2 was also tested by thermal stability with temperature range at 300-700 ^{0}C. The results showed that the FT-IR spectrum of B_2O_3-SiO_2 has some vibrations of B-O, Si-O-Si, Si-O-B stretching, and Si-O-B bending at 1442.8 cm^{-1}, 779.2 cm^{-1}, 925.8 cm^{-1}, and 648.1 cm^{-1}. The X-Ray diffraction pattern results showed that the analysis of B_2O_3-SiO_2 has high crystallinity with two peaks diffraction identified at 26.6^0 and 20.9^0. The thermal stability test of B_2O_3-SiO_2 showed that B_2O_3-SiO_2 has high thermal stability with temperature range at 300-700 ^{0}C. The results showed that the acidity analysis of B_2O_3-SiO_2 has potential number 122.71 mV so that indicated B_2O_3-SiO_2 was high acidity.

Keywords : boric oxide, silica dioxide, boric silica, B_2O_3-SiO_2

INTRODUCTION

The development of materials such as inorganic materials and hybrid organic inorganic materials is intriguing field as this decade due to applications of these materials for sensor, catalyst, ion exchange, membrane, and also for medical as drugs or agent transfer drugs. The development of these materials can be conducted using physical and chemical processes such as grafting (Chrouda et.al, 2015), sol-gel (McFarland and Opila, 2016), impregnation (Dhamodaran and Gnanaharan, 2007), support compound (Sari M A and Situngkir, 2016), and chemical reaction. In the chemical reaction, formation of chemical bonding is important in order to form stable novel compounds. Chemical reaction sometime involves high temperature and pressure thus this method is sometime omitted due to special reactor is required. In physical method such as impregnation and support method, electrostatic interaction, Van der Walls, or other physical interaction is formed on materials.

In recent years, support materials on inorganic matric is increased sharply. Inorganic substances such as boron compounds and its derivatives is interesting materials to develop due to high acidity resulted from non-bonding orbital of boron. Boron is found as various compounds in the nature such as boric acid and has been applied as catalyst (Kumar et.al, 2014). Boric acid has low thermal stability and the application of these compound in various field is still rare. To increase application of that compound thus modification of boric acid is vital. On the other sides, silica compounds such as silica oxide are frequently used in various application such as chromatography for separation and for polymer materials. Silica oxide can be used also as support material for inorganic matric (Jal, et.al 2004). Thus in this research silica oxide is used as support of boric compound. The final goal of this research is to obtain inorganic material with high acidity and stability under high temperature. High temperature stable material is needed for many applications such as catalyst and thin film (Moon et.al, 2004). The supported material is characterized using X-ray analysis, FTIR spectrophotometer, and acidity analysis by potentiometric titration.

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EXPERIMENTAL SECTION

Chemical with p.a. grade was used in this research from Merck such as boric acid, silica dioxide, acetonitrile, n-butylamine, ammonia, and buffer pH 4,7, and 10. Characterization was conducted using Shimadzu FTIR Prestige-21 with KBR disc, X-Ray Shimadzu Lab X type-6000 and the data was acquired over 0-90 deg. The acidity was analyzed by potentiometric titration using n-butylamine.

Preparation of B_2O_3-SiO_2

Boric acid (0.16 g) was dissolved with 30 mL water. The solution was stirring and heating at 80 ^{o}C for 10 minutes. Silica oxide (1.46 g) was added into the solution. The mixtures were refluxed for 5 hours at 90 ^{o}C. The mixtures was concentrated by vacuum to obtain white bulky solid. The solid material was heated at 110 ^{o}C for three days to form B_2O_3-SiO_2. Characterization of B_2O_3-SiO_2 was conducted using FTIR and X-Ray analyses.

Thermal Stability Test

Thermal stability test was conducted using muffle furnace and sample was heated for 3 hours at 400-700 ^{o}C. The material after heating process was analyzed by X-Ray analysis and acidity measurement.

RESULTS AND DISCUSSION

Material B_2O_3-SiO_2 was prepared via inorganic synthetic method without functional group protection. The objectives of this preparation is to obtain material with high acidity and high

stability under high temperature. Material B_2O_3-SiO_2 after synthesis was characterized using FTIR spectroscopy and FTIR spectrum is shown in Figure 1.

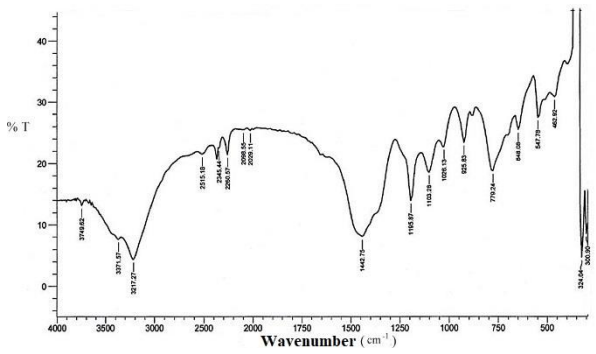

Figure 1. FTIR spectra of B_2O_3-SiO_2.

FTIR spectrum of B_2O_3-SiO_2 has vibration at wavenumber 3434 cm^{-1} (Si-OH), and 3227 cm^{-1} (B-OH). Stretching vibration of Si-O-Si is appeared at wavenumber 1091-800 cm^{-1} (Arkles, 1987). Vibration peaks at 927 cm^{-1} and 649 cm^{-1} are assigned as bending and stretching of Si-O-B and 1435 cm^{-1} is assigned as stretching of B-O. Identification of B_2O_3-SiO_2 is continued using X-ray analysis. The XRD powder pattern of B_2O_3-SiO_2 is shown in Figure 2.

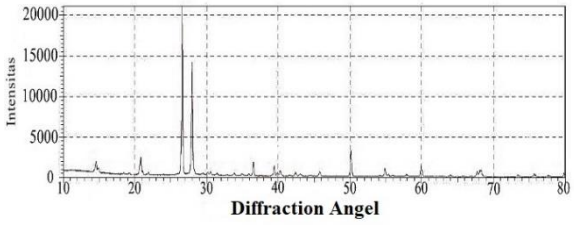

Figure 2. XRD powder pattern of B_2O_3-SiO_2.

Compound B_2O_3 has diffraction at 2θ value 26-27 deg and compound SiO_2 has diffraction at 2θ value 20-22 deg. Figure 2 show the diffraction at 2θ value 20 deg, 26 deg, and 29 deg, which is attributed to diffraction of B_2O_3-SiO_2 (Osiglio et.al, 2017). There is small diffraction at 2θ value 15 deg. Probably do to interaction of B_2O_3-SiO_2 and that diffraction indicated that material B_2O_3-SiO_2 has high crystallinity. The properties of B_2O_3-SiO_2 was studied by thermal stability test and acidity measurement.

The thermal stability test was conducted at 400-700 °C and the material was characterized using X-Ray analysis as shown in Figure 3. Diffraction patterns of XRD in Figure 3 showed that material B_2O_3-SiO_2 has high stability under high temperature. The pattern is almost similar each other after heating process. The patterns in Figure 3 is also fit with pattern in Figure 2. That means structure material B_2O_3-SiO_2 almost unchanged with increasing temperature.

The acidity material B_2O_3-SiO_2 and boric acid was tested using potentiometric titration as shown in Figure 4. The titration curve at figure 4 showed that boric acid has potential 57.17 mV and B_2O_3-SiO_2 has potential 122.71 mV. That results showed that material B_2O_3-SiO_2 has high acidity than boric acid. Thus material B_2O_3-SiO_2 is candidate for acid catalyst material.

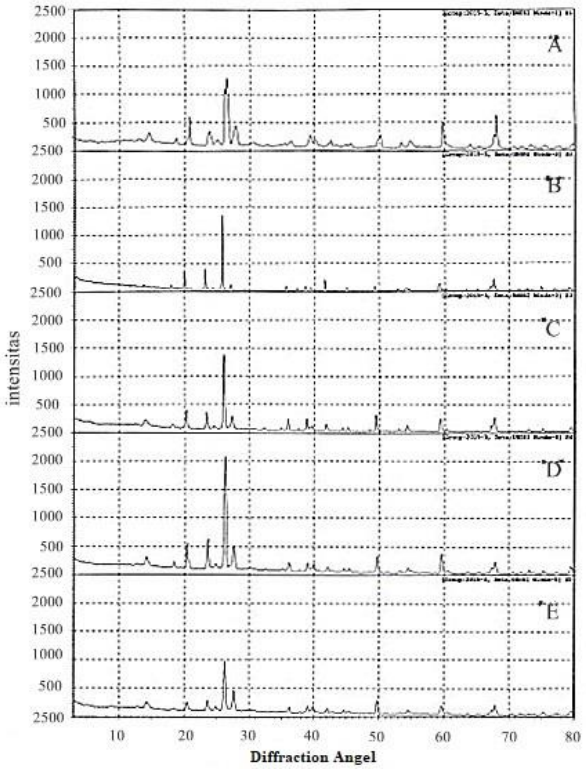

Figure 3. XRD powder patterns of B_2O_3-SiO_2 at various temperatures (A = 300 °C, B = 400 °C, C = 500 °C, D = 600 °C, E = 700 °C).

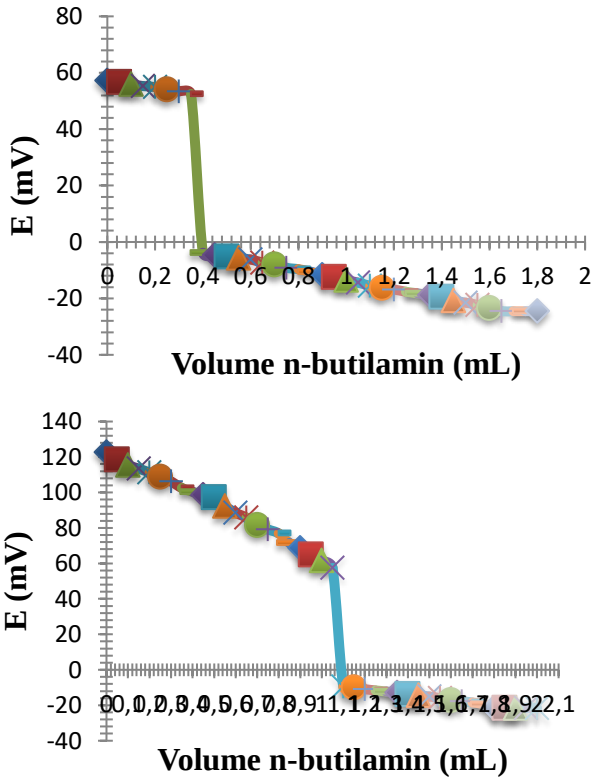

Figure 4. Potentiometric titration curve of boric acid (A) and B_2O_3-SiO_2 (B).

CONCLUSION

Material B_2O_3-SiO_2 was successfully synthesized with high crystallinity. This compound also has thermal stability and high Lewis acidity, which can be used as effective acid catalyst.

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