Removal of Adsorbed Moisture on Iron Powders by Rice Husk Ash

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Abstract

Rice husk ash was proposed to be used for removal of adsorbed moisture on iron powders to enhance their flow characteristic. It was produced by burning of rice husk at 700°C for 1 h, using a slowly increasing of heating rate (5°C/min). The burning condition was determined from thermal gravitation analysis of rice husk. The obtained rice husk ash contained 99.7% amorphous silica with large specific surface area of 295 m^2/g. Rice husk ash was effective in removal of adsorbed moisture on iron powders. Adsorption characteristic was found to be isothermal monolayer adsorption. The treated iron powders can be compacted more efficiently, obtaining green compact at a density higher than the untreated iron powders. Sintered billets of green compacts of treated iron powders showed a greater degree of particle bonding, having higher density and less fraction of pores.

Key words : Rice husk ash, adsorption, relative density, humidity, BET, iron, powder, pore.

Introduction

In many industries where raw materials are used in form of powders such as food, pharmaceutical, and powder metallurgy industries, a major processing step is controlled flow of powders. Any unpredictability of flow behavior of powders will usually result in inhomogeneity of final products. Flowability of powders is known to be affected by various kinds of adhesion forces such as Van der Waals, electrostatic, solid bridge, and liquid bridge or capillary forces. At high ambient humidity level, however, the largest adhesion force is identified to be capillary force.[1] Capillary force is created by liquid condensation due to adsorbed moisture on powder surface. At high humidity level, liquid bridge between adjacent powders is formed and results in capillary force. Capillary force causes agglomeration of powders and acts against flow of powder; either it is a forced flow or free flow under gravity.[2]

Powder metallurgy process involves free flow of metal powders into a die cavity before compaction at room temperature to form a near-net-shape product. The compacted powder, the so-called green compact, is subsequently sintered at high temperature to remove pores and produce near full-density metal parts. The density of final products critically relates to the density of the green compact, which in turn is affected by the ability of metal powders to flow and rearrange in die cavity.[3]

In this study, rice husk ash, which is known for its ability as adsorbent,[4,5] is used to remove moisture adsorbed on iron powders. The effect of moisture removal was investigated by comparison between final products produced from iron powders which are treated and untreated by rice husk ash. Relative density of green compacts and microstructure of final products were analysed.

Materials and Experimental Procedure

Preparation of Rice Husk Ash

Rice husk from Lopburi province of Thailand was selected to be studied for its ability to adsorb moisture. Rice husk was first treated by 3 NHCl to remove metallic impurities.[6] Thermal characteristic of the acid leached rice husk was characterized by The thermal Gravitational Analyzer (TGA, Netzsch STA 409). From the

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TGA result, the burning temperature was selected as 700°C for 1 h in air, using a low heating rate of 5°C/min. Surface morphology of the obtained rice husk ash (RHA) was studied by SEM. Chemical composition and phases of RHA were analyzed by X-ray fluorescence (XRF) and X-ray diffraction (XRD) techniques, respectively. Specific surface area was analyzed by BET method.

Removal of Adsorbed Moisture on Iron Powders by RHA

The ability of RHA to adsorb moisture was studied by placing 25, 50, and 75 grams of RHA in 600 ml beakers, each to be treated with 20 grams of iron powders. Iron powders were commercial grade of 99.8% purity and with a mean particle size of 110 μm. Ability to adsorb moisture of RHA was compared with that of commercially available silica gel by placing 25 grams of RHA and silica gel in 600 ml beakers, each with 20 grams of iron powders. Before use in each experiment, RHA and silica gel were baked in an oven at 110°C for 6 h in air to remove any adsorbed moisture, and quickly transferred into the beakers. Relative humidity level of the air inside the beakers was monitored by a handheld hygrometer (Custom CTS350).

Compaction and Sintering of Treated Iron Powders

After removal of moisture by RHA, three grams of treated iron powders were transferred to chambers which had 20% relative humidity. The powders were compacted inside the chamber under 207 MPa pressure to make a 10 mm diameter green compact. The density of the green compacts was measured. The green compacts were subsequently sintered at 1050°C for 1 h in flow of argon; heating rate to that temperature was increased by 10°C/min. The microstructure of sintered specimen was investigated by optical microscope.

Results and Discussion

Rice husk is composed of cellulose and metallic elements commonly found in minerals and soil.[6] In order to obtain pure white silica, it must be acid leached and burnt to remove impurities and cellulose. The thermal characteristic of rice husk used in this experiment is shown in Figure 1. Upon heating, mass loss is continuous with a broad peak in the 130 to 350°C range. Starting at 700°C, there is no longer any mass loss. This means that thermal

decomposition of rice husk stops at a temperature around 700°C; therefore, the burning condition is selected as 700°C to ascertain complete removal of cellulose and impurities. The heating rate to the temperature is selected as 5°C/min to slowly burn off the cellulose. Figure 2 shows the surface morphology of the rice husk ash (RHA) obtained. The surface is rough with deep crevices, resembling surface morphology of rice husk. Specific surface area is as high as 295 m^2/g. Chemical composition analysis by XRF is shown in Table 1. RHA is composed of 99.7% silica with trace amount of oxides of potassium, calcium, phosphorous, aluminum, and iron. The X-ray diffraction pattern of RHA in Figure 3 contains a broad peak at 22.5 degree, which is typical of amorphous silica.[6] Detailed investigation of burning of rice husk to obtain high purity white silica can be found elsewhere.[6-8]
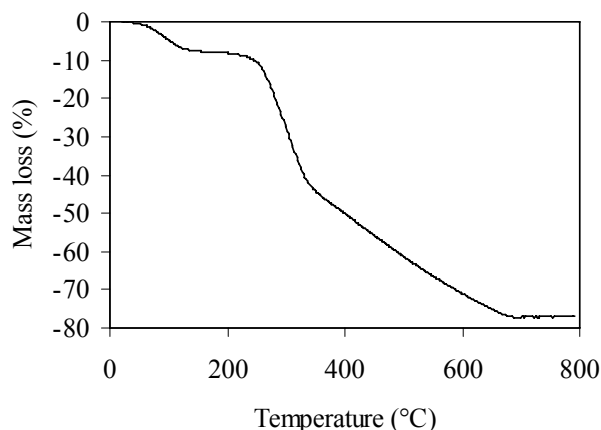
Figure 1. Thermal gravitational analysis of rice husk.
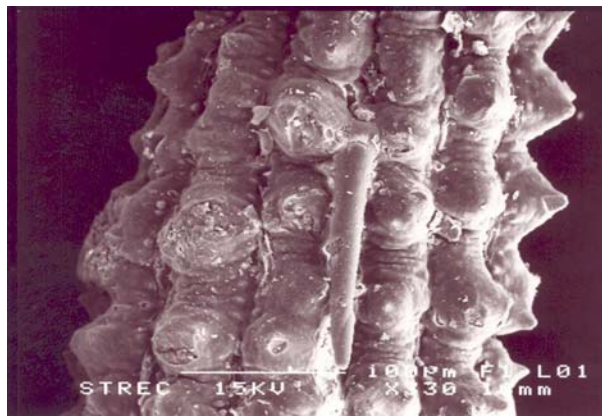
Figure 2. Scanning electron micrograph of rice husk ash.
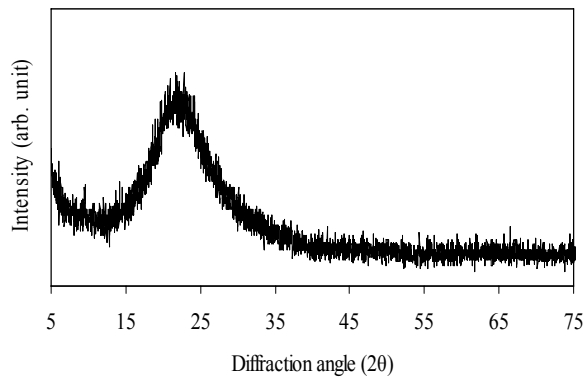
Figure 3. X-ray diffraction pattern of rice husk ash.

Table 1. Chemical composition of rice hush ash.

| Compound	Weight %
SiO_2	99.693
Al_2O_3	0.104
P_2O_5	0.037
K_2O	0.056
CaO	0.074
Fe_2O_3	0.036

It is known that RHA is a good adsorbent for heavy metal ions from aqueous solution.[4,5] In this study, RHA is used to adsorb moisture in a sealed 600 ml beaker with 20 grams of iron powders to be treated. Figure 4 shows the change of relative humidity level in the sealed beaker after placing different amounts of RHA inside. The change of relative humidity for different amounts of RHA used shows the same trend. The relative humidity level decreases quickly at the beginning, and levels off to about 20% decreases at long time. In addition, larger amount of RHA can remove moisture faster, and thus reaches a lower level of relative humidity. Figure 4 also shows the percentage removal of moisture inside the beaker. It is calculated from the difference between the instantaneous relative humidity level and the starting relative humidity level, divided by the starting relative humidity level. This reflects adsorption of moisture vapor by RHA. Adsorption is fast at the beginning, which can be seen by the high slope of the curves, and becomes slow at long time as the curves become flat.

Classically, adsorption phenomenon is explained by the isothermal multilayer adsorption model (BET model).[9] It is a generalization of Langmuir's isothermal monolayer adsorption model.

According to BET model, the total volume (v) of adsorbed moisture varies with relative humidity (x) as

$$\frac{v}{v_m} = \frac{cx}{(1-x)[1+(c-1)x]} \, ,$$

where v_m is the volume of the first layer of adsorption and c is a constant for an interface and temperature during adsorption. Note that relative humidity is the partial pressure of moisture divided by the saturated partial pressure of moisture at the temperature. Typical relationship between v/v_m and x for $c = 100$ is shown in Figure 5, which represents the equilibrium relationship between volume of gas adsorbed on a surface at different partial pressure of the gas.
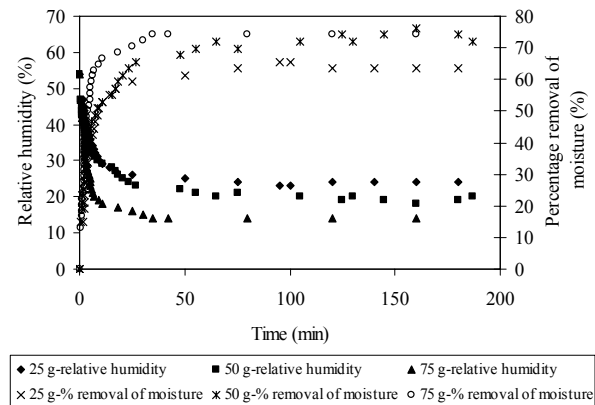
Figure 4. Adsorption of moisture by different amount of rice husk ash in a 600 ml beaker containing 20 g of iron powders.
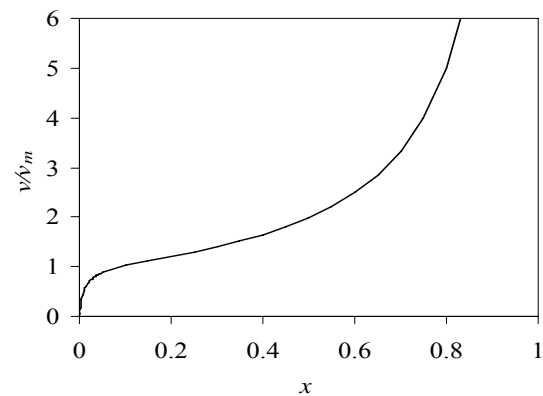

Figure 5. Isothermal multilayer adsorption model (BET model) showing equilibrium adsorbed volume (v/v_m) at different relative humidity levels (x).

Change of humidity level inside the beakers can be explained by adsorption of moisture vapor by RHA. The curves in Figure 4 for the percentage removal of moisture inside the beaker, which is the adsorption of moisture vapor by RHA, resemble the adsorption characteristic of the BET

model, in the region where v/v_m is less than one. In other words, it is a monolayer adsorption phenomenon during which the first monolayer of adsorbate is formed, and the total volume of adsorbate is less than the volume of the first single layer of adsorbate. The adsorption characteristic of RHA shown in Figure 4 characterizes formation of the first single layer of moisture vapor molecules on RHA surfaces. At the beginning, where most RHA surfaces are clean and available, moisture can be adsorbed onto RHA easily, and the adsorption rate is high. At this stage, relative humidity in the beaker decreases quickly.

As the adsorption process continues, adsorption rate becomes slow as most surfaces of RHA become covered with moisture vapor molecules. Relative humidity in the beaker decreases at a slower rate. Finally, when the surfaces are completely covered with a single layer of moisture vapor molecule, adsorption process stops, and relative humidity level in the beaker becomes constant. The formation of a monolayer of moisture vapor molecules on RHA surfaces is completed. Since adsorption is a surface phenomenon, larger amounts of RHA with larger total surface area is more effective in adsorption; therefore, a faster adsorption rate is observed when placing a larger amount of RHA in the beaker.

Figure 6 shows the comparison of adsorption ability of RHA and silica gel. It can be seen that RHA posses abilities comparable to the commercially available silica gel. The adsorption characteristic of both materials is monolayer adsorption, as explained earlier. By using RHA, the final relative humidity in the 600 ml beaker is only 20% for within just 50 minutes.
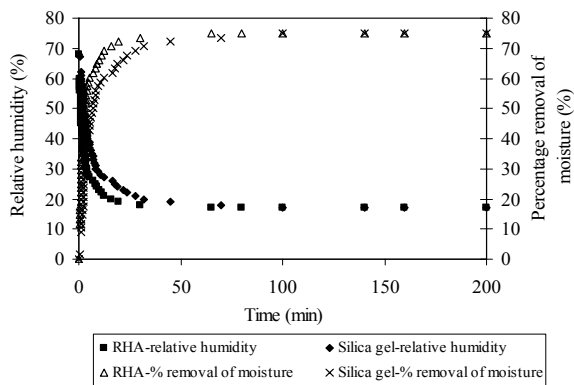
Figure 6. Comparison of ability to adsorb moisture of rice husk ash and silica gel in 600 ml beakers containing 20 g of iron powders.
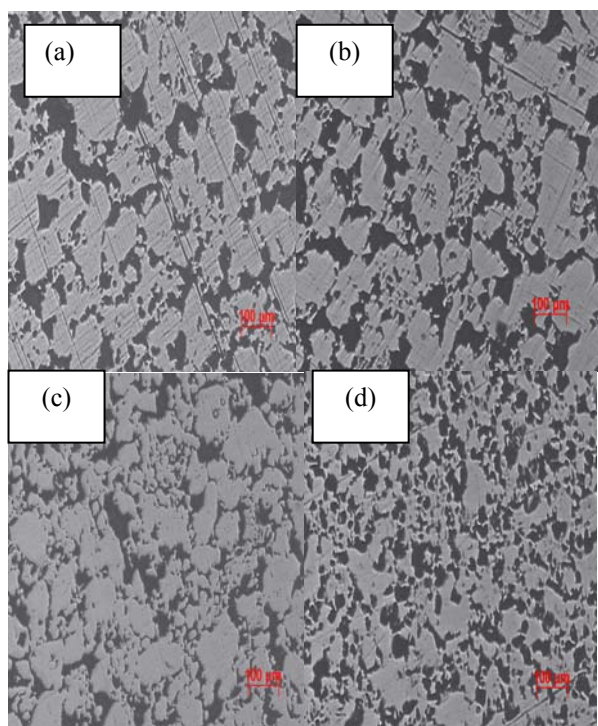
Density of green compacts and sintered billets made from compaction of treated iron powder (20% relative humidity) is shown in Figure 7. The figure also shows densities of green compacts at 45, 75, and 98% humidity levels. The density of green compacts and sintered billets is humidity dependent. When compactions are done at low humidity level, relative densities of green compacts are in the 73 to 74% range. Compaction at high humidity level reduces their relative density to less than 70%. Note that, in general, the relative density values are small due to the low applied compaction load of 207 MPa. Moisture in untreated powder creates adhesion between particles by capillary force.[2] Adhesion between powder particles deteriorates flow property of powder particles, as it prevents sliding and rearranging of particles. Therefore, density of green compact obtained is lower for compaction at high relative humidity level. After sintering, densities increase slightly. The amount of density increase is highest for the sintered billet of 98% humidity level; although, the obtained density is less than those of sintered billets of lower humidity levels.
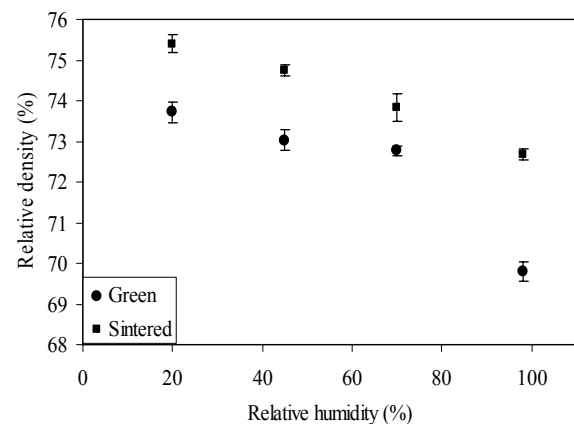
Figure 7. Relative density of sintered billet and green compacts which were compacted under different humidity levels.

The structures of pores in sintered billets are shown in Figure 8. Figures 8 (a) to 8 (d) are microstructures of sintered billets made from compaction under 20, 45, 75, and 98 percent humidity, respectively. Image analysis software reveals that the fraction of pore areas in Figures 8 (a) to 8 (d) is 32, 35, 31, and 40 percent, respectively. The pore fraction values show the same trend as the density measurements in Figure 7; i.e., compaction at high relative humidity results

in low relative density in both green compact and sintered billet, in which a large amount of pores is still present. By comparison between Figure 8 (a) and 8 (d), the former shows a greater degree of bonding between particles with interconnected large pores, while the later shows a smaller degree of bonding between particles with interconnected small pores. Moisture in untreated powders may cause agglomeration of powders which affects sintering behavior of green compacts. Sintering of green compacts which are compacted at low relative humidity level, yields a better degree of particle bonding than sintering of green compacts, which are compacted at high relative humidity level.

Figure 8. Microstructure of sintered billets produced from green compacts at different humidity levels; (a) 20%, (b) 45%, (c) 75%, (d) 98%.

Conclusions

Burning of rice husk at 700°C using a slow heating rate of 5°C/min can produce rice husk ash which is 99.7% purity silica. The obtained silica is amorphous with a large specific surface area, able to adsorb moisture from iron powders. The moisture adsorption ability is comparable to that of commercially available silica gel. The adsorption characteristic was found to be isothermal monolayer adsorption. The effect of removal of moisture from iron powders is a density increase of green compacts and sintered billets. Sintering of green compacts made from treated iron powders gives a greater degree of particle bonding and results in a higher density with lower fraction of pore area.

Acknowledgement

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