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Apparent Porosity and Compressive Strength of Heat-Treated Clay/Iron Sand/Rice Husk Ash Composites over a Range of Sintering Temperatures

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Abstract

Novel composites of clay/iron sand/rice husk ash (RHA) have been developed. Electric furnace was used to perform heat treatment on the composites to study the effect of sintering temperature on their apparent porosity and compressive strength. Two types of RHA with different bulk density were prepared to gain an understanding of the influence of apparent porosity on compressive strength of the heat-treated composites over a range of sintering temperatures. Heat-treated composites, made of clay/iron sand and clay/RHA, were also prepared as a referenced material. X-ray diffraction (XRD) analysis was further performed to comprehensively discuss the role of iron sand on apparent porosity and compressive strength of the heat-treated composites. The results show that the increase of sintering temperature reduces apparent porosity of the heat-treated composites. Reducing on the apparent porosity was then followed by the increase of compressive strength of the heat-treated composites. Compressive strength of the heat-treated composites was not sensitive to the sintering temperature up to 800 °C, and it would be more improved at the sintering temperature above 800 °C. This study concludes that such sintering temperature significantly improved apparent porosity and compressive strength of the composites due to use of iron sand.

Abstrak

Porositas dan Kuat Tekan dari Komposit Tanah Liat/Pasir Besi/Abu Sekam Padi (ASP) Setelah Menerima Perlakuan Panas pada Beberapa Rentang Temperatur Sintering. Komposit baru yang terdiri atas tanah liat/pasir besi/abu sekam padi (RHA- *Rice Husk Ash*) telah dikembangkan. Tungku elektrik digunakan untuk memberikan panas pada komposit untuk mempelajari efek suhu pengerasan pada porositas yang nyata dan kekuatan tekan dari komposit tersebut. Dua tipe RHA dengan bobot isi berbeda dipersiapkan untuk mendapatkan pemahaman akan pengaruh porositas yang nyata pada kekuatan tekan dari komposit yang diberi panas berdasarkan kisaran suhu pengerasan. Komposit yang diberikan panas, yang terbuat dari tanah liat/pasir besi dan tanah liat/RHA, juga dipersiapkan sebagai materi yang diacu. Analisis XRD (*X-ray diffraction* – difraksi X-ray) selanjutnya dilakukan untuk secara komprehensif membahas peran pasir besi pada porositas yang nyata dan kekuatan tekan dari komposit yang diberi panas. Hasilnya menunjukkan bahwa peningkatan suhu pengerasan mengurangi porositas yang nyata dari komposit yang diberi panas. Pengurangan pada porositas yang nyata itu kemudian diikuti oleh peningkatan kekuatan tekan dari komposit yang diberi panas. Kekuatan tekan dari komposit yang diberi panas tidaklah peka terhadap suhu pengerasan sampai pada 800 °C, dan keadaan akan lebih baik pada suhu pengerasan di atas 800 °C. Penelitian ini menyimpulkan bahwa suhu pengerasan di atas 800 °C secara signifikan memperbaiki porositas yang nyata dan kekuatan tekan dari komposit karena penggunaan pasir besi.

Keywords: clay, composites, iron sand, RHA, sintering temperature

1. Introduction

Nowadays, a variety of materials and structures made of clay composites has been found in many modern applications of engineering [1]. Mixing clay and other materials has even been an alternative route to design a set of composite materials with desired properties [2-4]. Partial replacement of clay-sand and lateritic soil-clay

by Rice Husk Ash (RHA), for instance, has not only increased compressive strength [5-6] but also significantly improved linear shrinkage and bulk density of clay bricks [7].

For a particular purpose, Machmud [8] and Machmud *et al.* [9] proposed a set of heated clay/marine sand/RHA composites in a preliminary study on temperature sensitivity of compressive strength of the composites because of partial replacement of marine sand with RHA. In the later study, it is pointed out that compressive strength of the composites was significantly improved at the temperatures above 850 °C. At 900 °C and 1000 °C, the best compressive strength was achieved for 20%wt and 30%wt RHA, respectively. In the other study, Machmud *et al.* [10] reveal that although such composite materials are potential for the purpose, the use of the marine sand is not recommended due to Cl^- and SO_4^- ions contents and many other lacks due to environmental concerns.

Due to such lacks of the marine sand, iron sand, a natural resource which has been bountifully available in Aceh, Indonesia, was further used to produce better novel clay composite instead. Nevertheless, the use of the iron sand should be maintained as minimum as possible for a long-term sustainable plan. For this reason, partial replacement of the iron sand with RHA, an agricultural waste by-product which is also abundantly available in Aceh, Indonesia, has been applied in this present study to propose a set of clay/iron sand/RHA composites. Two types of RHA with different bulk density were used. Through partial replacement of the iron sand with a certain weight fraction of such RHA, this paper has been prepared to gain an understanding of influence of the apparent porosity on compressive strength of clay/iron sand/RHA composites over a range of sintering tem-peratures.

2. Methods

The clay, iron sand, and RHA were from Blang Bintang, Lam Panah, and Darussalam, Aceh Besar, Indonesia, respectively. Two types of RHA which are further called as RHA1 with a bulk density of 0.444 g/cm^3 and RHA2 with a bulk density of 0.317 g/cm^3 are used. Chemical compositions of the raw materials were characterized by using X-ray fluorescence (XRF, Bruker S2 Ranger) and they are then listed in Table 1. There are no significant differences between RHA1 and RHA2. Iron sand has the lowest content of SiO_2 (0.58%wt). The contents of SiO_2 in both RHA materials (RHA1: 92.40%wt and RHA2: 92.22%wt) are higher than those in both clay (54.48%wt) and iron sand (0.58%wt), but the contents of Al_2O_3 and Fe_2O_3 in clay (16.77%wt and 15.48%wt, respectively) are the highest.

A number of processes, such as crushing, mixing, stirring, moisturizing, grinding, pasta forming, rack drying, and finally open-air drying were respectively performed to prepare the composites. In this study, the crushing process to obtain finer sizes was only performed on clay. Selecting size of the clay, iron sand and RHA particles were then performed through a sieve method using the US Standard Sieve No. 70 (0.210 mm). At the mixing process, a mixture of clay and iron sand was respectively set to a volume ratio 3:1. The stirring process was then performed to ensure an even distribution of the iron sand in the mixture. Water content up to optimum moisture content was further gradually added to the mixture of clay and iron sand. This process finished after taking time for 12 hours. The grinding process was then performed for 2 hours. Pasta forming and the set of drying processes completely turned the mixture then to a clay/iron sand composite. Using the same preparation method, composites of clay/iron sand/RHA were separately prepared by replacing 20% of the iron sand weight with each of RHA1 and RHA2. Composite samples of clay/iron sand/ RHA1 and clay/iron sand/RHA2 were finally prepared in a cubic shape of 70 mm × 70 mm × 70 mm. Composite samples of clay/iron sand, clay/RHA1 and clay/RHA2 were also prepared as referenced materials.

For every 1500 g of the clay, it was found that the water content required by each of the composites to achieve optimum moisture content was different. The water

Table 1. Chemical Compositions of Raw Materials (%wt)

Composition	*Clay*	Iron Sand	RHA1	RHA2
SiO_2	54.48	0.58	92.4	92.22
Fe_3O_4	-	86.55	-	-
Al_2O_3	16.77	0.82	0.74	0.78
Fe_2O_3	15.48	-	0.28	0.14
MgO	3.74	-	-	-
CaO	3.44	0.40	0.77	0.78
K_2O	2.23	0.30	2.73	2.71
TiO_2	1.55	6.10	-	-
P_2O_5	0.69	0.40	1.73	1.64
SO_3	0.58	0.16	0.61	0.95
MnO	0.14	0.74	0.17	0.13
Cr_2O_3	0.11	2.01	0.01	-
SrO	0.08	-	-	-
Pr_6O_{11}	-	0.40	-	-
Nd_2O_3	0.07	0.33	-	-
ZrO_2	0.06	0.02	-	-
NiO	0.03	0.62	-	-
SnO_2	0.03	0.02	-	-
ZnO	0.03	0.06	-	0.02
V_2O_5	0.02	-	-	-
CuO	0.02	0.08	-	-
Rb_2O	0.01	-	0.02	0.01
MoO_3	-	0.03	-	-
CoO	-	0.25		
La_2O_3	-	-	-	0.02
Others	0.39	0.20	0.46	0.58

content required by each of the composites for every 1500 g of the clay is shown in Figure 1. Figure 1 shows that partial replacement of the iron sand with RHA in the mixtures increases the content of water. It also shows that water absorption of RHA2 (0.317 g/cm^3) is higher than that of RHA1 (0.444 g/cm^3).

A set of heat treatment on the composite samples was then performed using an electric furnace to prepare the heat-treated composite (HTC) materials. Prior to the heat treatment, however, each of the open-air dried composite samples gradually re-dried up to 110 ± 5 °C for 48 h using a ventilated oven. Such drying was then repeated and finally stopped until the mass of the samples remained constant. The heat treatment was then performed for 700 °C, 800 °C, and 900 °C as a set of sintering temperatures. The samples were firstly heated from room temperature (30 °C) to 200 °C at a constant rate of 1 °C/min and holding time for 4 h. The latter temperature was subsequently elevated to 500 °C at a constant rate of 3 °C/min and holding time for 4 h. Each of the samples was finally heated at 700 °C, 800 °C, and 900 °C at a constant rate of 5 °C/min and holding time for 12 h to properly sinter the HTC samples before cooling the sintering temperature down to room temperature (30 °C). The rate of temperature for cooling the composites was not set. Such heat treatment program is schematically described in Figure 2. Furthermore, CIA-11, CIA-12, and CIA-13 are presented as initials of the heat-treated clay/iron sand/RHA1 composite samples heated at 700 °C, 800 °C and 900 °C, respectively. However, CIA-21, CIA-22, and CIA-23 are presented as initials of the heat-treated clay/iron sand/RHA2 composites heated at 700 °C, 800 °C and 900 °C, respectively. Each of HTC of clay/iron sand, clay/RHA1 and clay/RHA2 was prepared as referenced materials. Hence, CIX-01, CIX-02, and CIX-03 are also presented as initials of the HTC

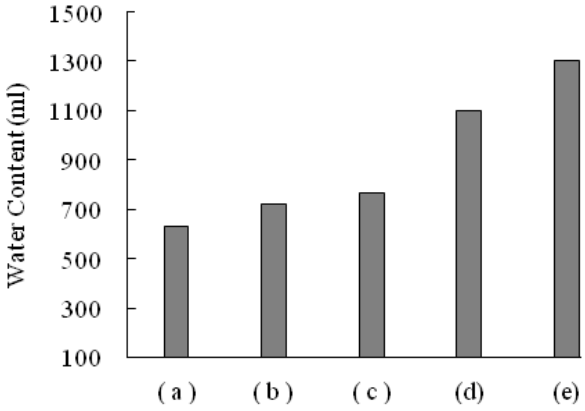

Figure 1. Water Content (ml) Required by Each Composite: (a) Clay/Iron Sand (b) Clay/Iron Sand/RHA1 (c) Clay/Iron Sand/RHA2 (d) Clay/RHA1 and (e) Clay/RHA2

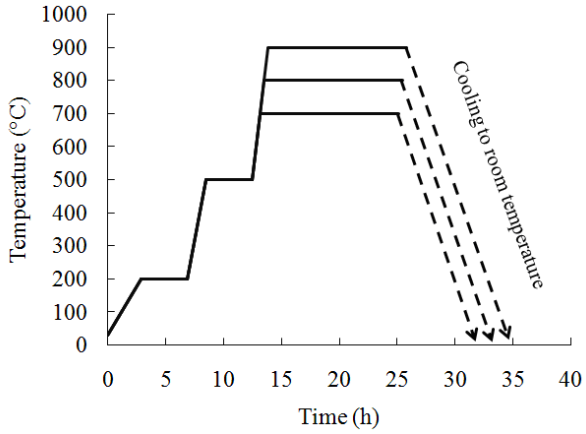

Figure 2. Heat Treatment Programs of the Composite Samples over a Range of Sintering Temperatures

of clay/iron sand heated at 700 °C, 800 °C and 900 °C, respectively. Similarly, CXA-11, CXA-12, and CXA-13 are presented as initials of the HTC of clay/RHA1 heated at 700 °C, 800 °C and 900 °C, respectively. Meanwhile, CXA-21, CXA-22, and CXA-23 are presented as initials of the heat-treated composites of clay/RHA2 heated at 700 °C, 800 °C and 900 °C, respectively.

The apparent porosity *AP* of the HTC materials was determined according to ASTM C20-00 [11]. It was calculated using Eq. (1) where *S*, *D*, and *W* are soaked, furnace dry and suspended states weight of the HTC samples, respectively. The soaked state weight *S* was measured after soaking the HTC samples into water for 24 h. Meanwhile, the suspended state weight of the HTC samples *W* was measured at room temperature after 5 h boiling the samples in water.

$$AP = \frac{S-D}{S-W} \times 1 \quad (1)$$

Bulk density of the HTC samples *BD* is also presented and it was calculated using Eq. (2).

$$BD = \frac{S}{S-W} \times \rho_{water} \quad (2)$$

A compression test in accordance with ASTM C67-08 [12] was carried out at a room temperature to determine compressive strength of the HTC materials. Such compression test was performed on HTC samples with a cubic shape of 50 × 50 × 50 (mm) using a universal testing machine (HT-2402, 250kN) at a constant rate of 20 mm/min.

After crushing a set of prepared HTC samples, powders of the samples were collected and passed through a sieve method using the US Standard Sieve No. 200 (0.075 mm). X-ray diffraction (XRD, the Shimadzu

D6000) analysis was further carried out on the powder in the scanning range of 10° to 80° (2θ) at a rate of 2° min^{-1} with a voltage of 40 kV and a constant current of 30mA to identify the crystallite phases of the HTC materials.

3. Results and Discussion

A list of apparent porosity and bulk density of the HTC materials is presented in Table 2. As it was designed, Table 2 shows that apparent porosity of clay/iron sand/RHA2 is higher than that of clay/iron sand/RHA1. Similarly, apparent porosity of clay/RHA2 is higher than that of clay/RHA1.

Table 2 indicates that RHA is responsible for the increase of the apparent porosity of the HTC materials. It is shown that the lowest apparent porosity is achieved by HTC made of the clay/iron sand. Bulk density of the latter composites is hence slightly higher than that of the other HTC materials. Table 2 also shows that heat treatment on the HTC materials over a range of sintering temperatures affects apparent porosity of HTC materials. Apparent porosity of the HTC materials reduced with increase of the sintering temperature.

Compressive strength of the HTC materials over a range of sintering temperatures is presented in Figure 3. It is shown that compressive strength exhibited by the HTC materials made of clay/iron sand is better than that of the other HTC materials at the whole range of sintering temperatures.

Figure 3 also shows that sintering temperature carried out up to 800 °C does not significantly affect compressive strength of the HTC materials, and different compressive strength at a similar sintering temperature was determined by their apparent porosity. Although apparent porosity was a bit better improved up to that point, compressive strength of each HTC was not improved very well. Compressive strength of the HTC materials made of both clay/RHA composites, however, was not even significantly improved in the whole range of the sintering temperatures. Compressive strength of the latter HTC materials was even very poor. Such results indicated that sintering temperature did not turn the HTC materials to

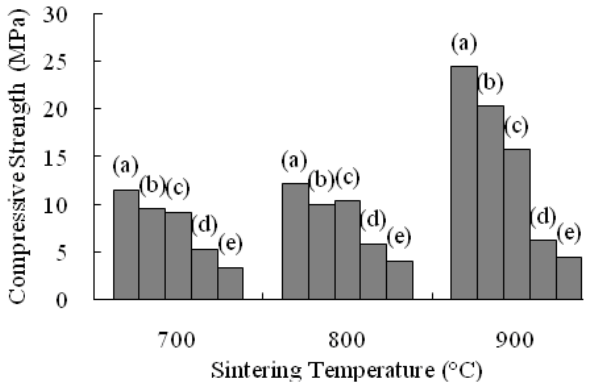

Figure 3. Compressive Strength of the HTC: (a) Clay/Iron Sand (b) Clay/Iron Sand/RHA1 (c) Clay/Iron Sand/RHA2 (d) Clay/RHA1 and (e) Clay/RHA2 Over a Range of Sintering Temperatures

Table 2. Apparent Porosity and Bulk Density of HTC Materials

HTC Materials	Sample Initials	T (° C)	Apparent porosity (%)	Bulk Density (g/cm^3)
Clay/Iron Sand	CIX-01	700	32.12	1.60
	CIX-02	800	27.20	1.65
	CIX-03	900	20.21	1.71
Clay/Iron Sand/RHA1	CIA-11	700	34.24	1.52
	CIA-12	800	31.46	1.61
	CIA-13	900	27.60	1.65
Clay/Iron Sand/RHA2	CIA-21	700	37.00	1.40
	CIA-22	800	34.44	1.52
	CIA-23	900	30.25	1.60
Clay/RHA1	CXA-11	700	39.60	1.35
	CXA-12	800	37.28	1.40
	CXA-13	900	33.08	1.55
Clay/RHA2	CXA-21	700	42.29	1.33
	CXA-22	800	40.47	1.36
	CXA-23	900	38.89	1.38

have a lower volume of pore. Their apparent porosities are still higher than those of the other HTC materials. The highly volume of pore in the HTC materials is because of fully replacement of the iron sand with RHA. This reason might cause compressive strength of the HTC materials made of both clay/RHA composites to become very poor. Fully replacement of the iron sand with RHA is therefore not recommended.

Generally, compressive strength of the HTC materials with the iron sand contents would be significantly improved at the range of sintering temperatures more than 800 °C. Such results, therefore, also indicate that the iron sand plays very important role not only on reducing apparent porosity but also on improvement of compressive strength of the HTC materials, i.e. sintering temperature has a significant effect on apparent porosity and compressive strength of the HTC materials at over 800 °C because of the content of the iron sand. This is in agreement with the results obtained from XRD analysis performed on the HTC made of clay/iron sand as presented in Figure 4a. Figure 4a shows that although most of Fe_3O_4 (the dominant phase in iron sand) transformed into Fe_2O_3 at 900 °C, the phase still existed in the material. The phase was not found any more in the HTC materials made of both clay/iron sand/RHA heated at 900 °C as presented in Figure 4b and 4c.

The XRD analysis performed on the HTC made of clay/RHA2, which was selected to represent a diffraction pattern of the HTC materials made of clay/RHA composites, as presented in Figure 4d, reveals that although this material is also composed of some dominant phases, such as SiO_2, Al_2O_3, and Fe_2O_3, the intensity of the SiO_2 phase contributed by both clay and RHA in this material is greater than that of the other HTC materials. Since SiO_2 contributed by both clay and RHA is in the amorphous phase, the greater phase of SiO_2 led to the increase of pore formation in the materials and it subsequently might cause the HTC materials made of clay/RHA to be brittle and have poor compressive strength.

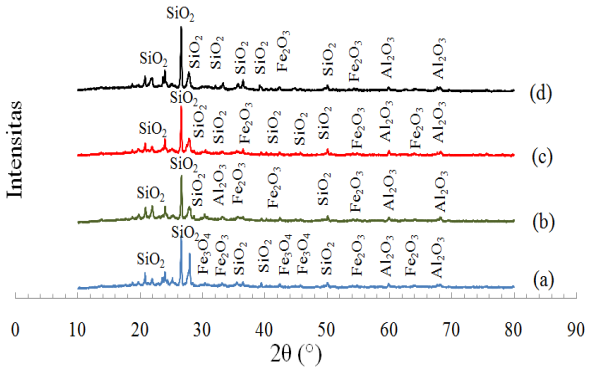

Figure 4. The XRD Diffraction Pattern of Heat-treated Composite at Sintering Temperature of 900 °C for: (a) Clay/Iron Sand, (b) Clay/Iron Sand/RHA1, (c) Clay/Iron Sand/RHA2 and (d) Clay/RHA2

4. Conclusions

A study to gain an understanding of influence of the apparent porosity on compressive strength of heat-treated clay/iron sand/RHA composites over a range of sintering temperatures has been presented. The study reveals that apparent porosity of the materials is affected by the applied sintering temperatures. Increase of sintering temperature leads to the decrease of volume of pore and subsequently increases the compressive strength of the HTC materials. However, improvement of apparent porosity because of the increase of sintering temperature does not significantly impact the compressive strength of the HTC materials made of clay/RHA. It is because of fully replacement of the iron sand with RHA. The phase of SiO_2 contributed by both clay and RHA is the amorphous phase, the greater phase of SiO_2; therefore, this leads to the increase of pore formation in the materials, and it thus subsequently might cause the HTC materials made of clay/RHA to be brittle and have poor compressive strength. Iron sand, dominantly composed of Fe_3O_4, plays a very important role on improvement of apparent porosity and compressive strength of the HTC materials. Apparent porosity and compressive strength of the HTC materials with the iron sand content will be significantly improved at the range of sintering temperatures more than 800 °C. The XRD analysis performed on the HTC materials points out that sintering temperature has a significant effect on compressive strength of the HTC materials at over 800 °C because of the contents of the iron sand.

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