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The Study of Porous Aggregate Manufacture with the Black Ash from Gasification of Rice Husk

[1]Jai-Houng Leu and [2]K.S. Chou
[1]General Education Center, Yu-Da University, No. 168, Shuefu Rd., Chao-Chiao
Miaoli,Taiwan, Republic of China
[2]Department of Chemical Engineering, National Tsing Hua University, No. 101,
Section 2, Kuang-Fu Road, Hsinchu, Taiwan 30013, Republic of China

Abstract: This study aimed to propose the processing conditions and formula of making porous aggregates with the black ash, which is produced from gasification of rice husk and to provide a new environmental protection material for industry. The results indicated that higher thermal temperature results in fewer impurities in this black ash, as well as better SiO_2 crystallization. Relatively, surface area reduced and water flow loss is decreased. The designed mixture of intensifier and binder added in the sintered porous aggregates can increase strength without causing great loss of surface areas. There is an optimal condition existing. The recommended components include kaolin, starch and this black ash, which ratio is 0.9:0.3:2 are treated by some dispersing agent ($CaCO_3$ solution) for the uniformity of this mixture and sintered at 900°C, with less oxygen for this optimal process. The surface area of the aggregate is 105.88.88 $m^2\ g^{-1}$, strength is 123.95 psia, and loss on water flow is 1.1277% in the optimal condition.

Key words: Porous aggregate, rice husk, sintering processes, additive, binder

INTRODUCTION

Chiang *et al*. (2008) study the pyrolysis kinetics of rice husk in different oxygen concentrations and reveal that the essential components of such black ash are SiO_2 and a small quantity of metallic oxide. The main metals contain Ca, Mg, Fe, Al, Cu, Na and Ti. Moreover, varying amount of fixed carbon is emitted from the black ash under conditions set by pyrolysis or gasification system.

Leu and Lo (2007) reported that the black ash can be manufactured as substitute (refuse derived fuel) of traditional solid coal through press temperature and stress with additive PVA -1000 in acid salt solution. The product possesses the compression strength. So, the black ash from rice husk gasification possesses the potential use for making strong material with appropriate porosity and strength. That is, it can be manufactured as lightweight porous aggregate applied in industry or water treatment.

For porous ceramics study, Okada *et al*. (2008) discussed that it can be prepared from mixtures of allophane and vermiculite by uniformly axial pressing at 40 MPa and heated at 600-800°C. The bulk densities of samples, the number of smaller pores, the compressive strength and the water absorption rates are correlated with thermal treatment and mixture rate between allophone and vermiculite contents.

For lightweight aggregates study, De Gennaro *et al*. (2007) reported that an evaluation of the zeolitized facies of campanian ignimbrite as raw material for the production of lightweight expanded aggregates. This industrial waste (dried polishing mud) contains SiC, a phase known to act as additive. All materials evidenced a decreasing density with temperature whereas the same parameter decreases with increasing industrial waste content. Among the three tested mixtures, it showed that there is the appropriate treatment condition to produce the strength enhancement, density reduction and grain size holding. The technical features of these lightweight expanded aggregates were comparable to some expanded clays, with similar grain size, commercialized in Italy. Tsai *et al*. (2006) investigated that amorphous SiO_2 and Al_2O_3 were added to sewage sludge ash to analyze the characteristic changes of the lightweight aggregates. The aggregates become lighter in weight by prolonging the sintering time and raising the sintering temperature (above 1060°C) with high proportion of SiO_2(44.89%). The results showed that Al_2O_3 raised the compression resistance and fly ash lowered the sintering temperature required. That is, the additive compositions can result in predictable characteristic of lightweight aggregates from sewage sludge ash.

As for thermal treatment condition of lightweight aggregates sintering, Fu (2009) studied the single

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Corresponding Author: Jai-Houng Leu, General Education Center, Yu-Da University, No. 168, Shuefu Rd., Chao-Chiao, Miaoli, Taiwan, Republic of China

reservoir sediments were prepared at different thermal treatment (sintering temperature) conditions only. The results show that the density and compressive strength decrease as sintering temperature is lengthen. The Bulk density and compressive strength decrease as sintering duration is lengthen. But, raising sintering temperature increases the amount and size of closed pores, as well as the connectivity among the pores. The heating rate does not significantly influence the density of the aggregates. The optimal conditions of preparing lightweight aggregates are sintering temperature 1200-1250°C and duration 30 min. The properties of aggregates prepared are bulk density 1.45-1.77 g cm^{-3} and water absorbing less than 1%.

As for the additive, Martinovic *et al.* (2006) analyzed chemical, mineralogical and structural properties of diatomite samples with the aim of predicting the filtration efficiency. Beer filtration tests proved diatomite to be satisfactory and not causing any degradation of filtration process or beer quality. Chang *et al.* (2010) reported that F-class fly ash can enhance the compressive strength, in contrast, the diatomite can keep the original porous structure. By SEM and MIP measurements, the porous rate decreases to 62.34% while sintering temperature is 1200-1270°C. The alternative quantity of fly ash increases to 20%, the compressive strength is enhanced by two times, but, the porous rate decreases to 52.94% lower. Chen (2008) studied the feasibility of utilizing oyster shells, rice husk Ash, Water Treatment Plant Sludge (WTPS) and Basic Oxygen Furnace slag (BOF slag) as cement raw materials. Using wastes as cement raw materials has no significant effect on the formation of C_3S and C_2S phases. Alite-rich cement which is produced by wastes contains 48.81% of C_3S phase, and the amount of C_2S phase in Belite-rich cement produced by wastes reaches 76.48% while sintering temperature is 1400°C. According to the experimental results, the utilization of oyster shells, rice husk ash, WTPS and BOF slag as cement raw materials to produce Alite-rich cement and Belite-cement is feasible. It reveals that additives possess the feasibility to improve the compressive strength and enhance porous rate in the same time while raising sintering temperature.

Base on the above study reviewing, there is a compromise conditions for porous aggregate manufacture for both improvement of the compressive strength and porous rate. This study aims to propose suitable processing conditions and formula to produce porous aggregate with the black ash from rice husk gasification power plant.

MATERIALS AND METHODS

The black ash in this study was generated from the rice husk gasification system (provided by Renewable Energy Technology Co., in Taiwan) at 800°C with less oxygen, sintering aid was kaolin made from Japan (produced by Asahi Chemical Industry), and binder was starch (produced by Taiwan Starch Corp.). The experiments were conduct in the factory of Renewable Energy Technology Co., in Yulin county and Yu Da university in Miaoli county, R.O.C. and some experimental raw data was analyzed in National Tsing Hua University, R.O.C. The experimental equipments and method are described as follows:

Experimental instruments and equipments:

- **Scale:** SHIMADZU, MODEL AY-120
- **Multivolume pycometer:** Miraqe, model SD-120L
- **Furnace:** Jida Machinery, made in Taiwan
- **Oven:** Jida Machinery, made in Taiwan
- **Surface area analyzer:** Micromeritics ASAP-2010
- **Particle size analyzer:** Photo LPA-3100 and 3000
- **XRD:** Shimadzu, model XD-5
- **SEM:** JEOL TSM-T100, gold plating for samples 500 sec before taking picture
- **Strength tester:** MTS 810 Material Test System + 458 system Electronics
- **Extrusion molding machine:** Refer to design of Chen and Cawley (1992). In the experiment, the raw materials are poured into a column mold, Instron crosshead is slowly placed into the mold entrance, and the entrance is sealed with rubber cover. A vacuum pump is used to draw out air from the raw material for 35 sec and make Instron crosshead more closer to wall and push Instron crosshead into, let its depth exceed air exhaust hole; when performing extrusion, the rubber cover shall be removed and Instron crosshead controls pressure and speed

Research method: The black ash is grinded by grinder for 30 min and screened by 100-mesh (149 μm) screen. The powder is heated in oxygen-deficient state (temperature rise is 5°C min and final temperature is maintained 2 h). Then variation in density and surface area is discussed. Then, the black ash, binder and sintering aid is mixed at certain ratio to produce mixed slurry, which is extruded to shape and sintered. The surface area, strength and loss on water flow of the product will be measured. Based on the former formula, the sintering aid content (cost) is reduced and test is conducted again for suitable formula. The relevant preparation and measurement is as follows:

Preparation and heat treatment of this black ash slurry: Figure 1 shows the preparation process of this black ash slurry. The prepared this black ash slurry is conveyed to furnace for heat treatment.

Measurement of density and porosity: Regarding operation of Multivolume pycnometer, helium gas enters into the tested chamber to obtain balance pressure value, and then, into the expansion chamber for another balance pressure value (Chang, 1993). The sample volume can be calculated through ideal gas theory. The density is calculated through weight divided by the volume.

In terms of the density, porosity can be calculated by the following equation

$$\text{Porosity for green body} = \frac{(V_{gb} - V_{gr})}{V_{gb}} = \frac{(\rho_{gr} - \rho_{gb})}{\rho_{gb}}$$

$$\text{Porosity for sintered product} = \frac{(V_{pb} - V_{pr})}{V_{pb}} = \frac{(\rho_{pr} - \rho_{pb})}{\rho_{pb}}$$

Measurement of surface area: In the adsorption method, the pores which diameter is 20-15 A use gas (especially helium gas) absorption, and this method requires long time test and complicated calculation. In recent years, automatic devices and computers have been used to calculate more accurate results, as compared to other methods. This experiment utilized ASP 2010 for sample analysis and surface area is calculated through computer.

Strength measurement of finished products: The strength tester used in this experiment is MTS 810 Material Test System with 458 system Electronics. The sample is cylindrical and it is placed in the middle of the two contacts in strength test and aligned and the test speed is 1 mm min^{-1}. The computation method is present as follows:

Compression strength of samples:

- C = F/A
- C = Compression strength (kgf cm^{-2})
- F = Total load for destructive test (kgf)
- A = bearing surface (cm^2)

Strength estimate (take K: S: R=1:0:3:2 for example): Stacking density of finished products: 0.341±0.023 (m g^{-2}) (average of five measured values),

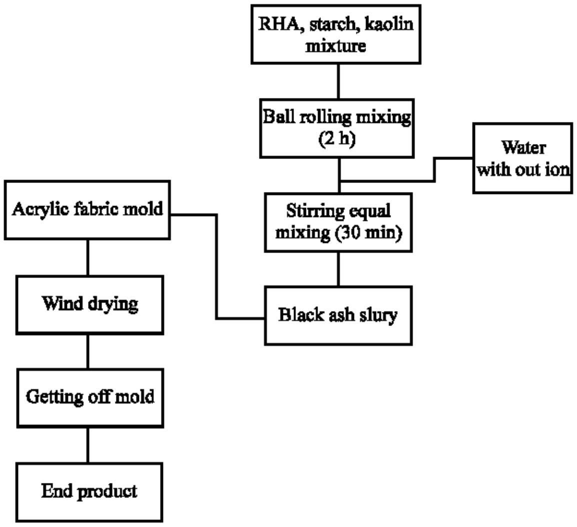

Fig. 1: The preparation process of this black ash slurry

- 1000 cm^3 samples with water is 1143.72 g
- If column height is 100 cm and diameter is 30 cm, column volume is $\frac{1}{4}\pi D^2 = (1/4)\pi(30^2)\times 100 = 70650$ cm^3
- The column is fully filled with samples and water, the total weight 7650/1000×1143.72 = 80803 g = 80.803 g Gravity of bottom is 80.803×9.8 = 791.87 kgf
- Area of column bottom is 706.5 cm^2
- Pressure of bottom calcinations is 791.87/706.5 = 1012 kgf = 15.95 psig = 29.65 psia
- It is assumed to be 1.5 times of safe pressure and safe pressure is 29.65×1.5 = 44.48 psia

Measurement of product surface washout amount: In the test, the samples are put into a cylindrical vessel, and the water is poured into the vessel from bottom to top (water flow rate is 4 L min^{-1}). The water should overflow from the vessel top after the samples are eroded. Accordingly, the test records the relationship between weight loss and wash time to understand loss of samples after fluid erosion.

RESULTS AND DISCUSSION

Heat treatment test: The heat treatment was carried out up to 1100°C in air and less-oxygen state separately. The original cristobalite faces of this ash are shown in Fig. 2 by XRD and SEM analysis. The carbon content smoothes the spectrum and the obvious porous structure enhance surface area ratio. It shows that the cristobalite faces of this ash toward pure SiO_2 cristobalite faces while the temperature attains 500°C in Fig. 3. The impurities of the black ash are volatized or burnt out due to higher temperature treatment. It also results in great loss of

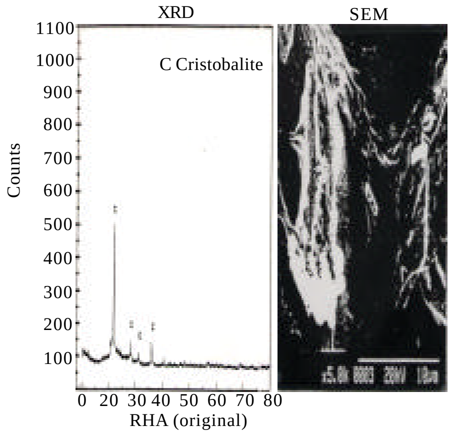

Fig. 2: XRD scan and SEM (5000X) analysis for original ash of rice husk

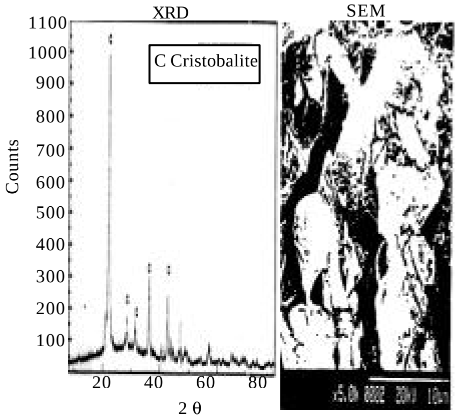

Fig. 3: XRD and SEM (5000X) analysis for the ash of rice husk after 500°C treatment

surface area. Tsai *et al*. (2006) reported that the aggregates become lighter in weight with high proportion of SiO_2 additive and raising the sintering temperature (above 106°C). The inherent SiO_2 content in this ash from rice husk has its effect obviously.

In the less-oxygen state, the black ash is put into a vessel and put the cover on the vessel to reduce contact between the black ashes, and keep out the air. It is evidenced the heat treatment effect is postponed by the less-oxygen state due to lean-air incineration. At temperature of 700°C, the density and surface area of the products reduce and the variation is very gentle, shown in Fig. 4. After exceeding 700°C, the variation is not great (between 2.25-2.29%), because the impurities are burnt out and the residue is approaching pure SiO_2 cristobalite faces.

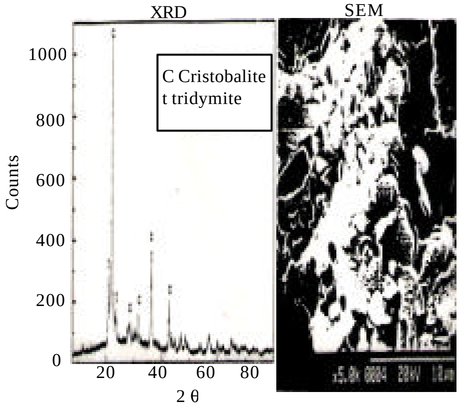

Fig. 4: XRD and SEM (5000X) analysis for the ash of rice husk after 700°C treatment (less-oxygen state)

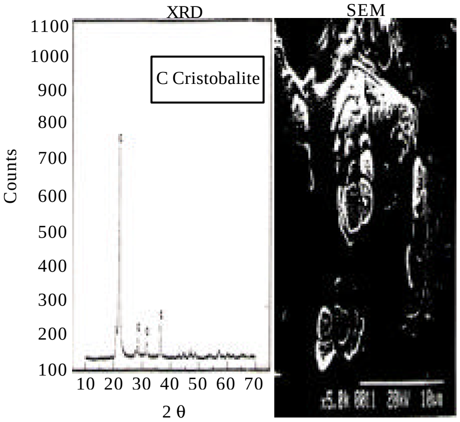

Fig. 5: XRD and SEM (5000X) analysis for the aggregates (kaolin: starch: ash=1.3:0.3:2) after 1100°C treatment (less-oxygen state)

Figure 5 reveals that no phase transformations of pure SiO_2 cristobalite faces occur until reaching the temperature of 1100°C for the mixture (kaolin: ash = 1.3:0.3:2) of the ash and the additive in this paper. It is concluded that the additives can postpone the form of pure SiO_2 cristobalite faces with raising the sintering temperature. That is, it enhances (or keeps) the surface rate when the strength of the aggregates may be improved by the sintering temperature. Fu (2009) studied the sintering thermal treatment conditions of light aggregates by reservoir sediments (impurities mixture) up to 1200-1250°C for achieving the structure strength. These trends of sintering thermal treatment with mixture are similar to the one in this study.

Black ash (R), starch (S) and kaolin (K) are mixed at different ratio (weight ratio) and sintered in the furnace as

Table 1: Property analysis for sintering conditions of different formulas at 1000°C

			BET					
			Surface area		Strength		Porosity	
Kaolin weight ratio (K)	Starch weight ratio (S)	Rice hull ash weight ratio (R)	$m^2 g^{-1}$	SD	psia	SD	%	SD
1	0.8	2	3.97	0.001	90.05	14.30	68	2.3
		3	3.82	0.002	93.48	8.23	62	3.5
		4	3.64	0.003	96.55	17.52	57	4.1
	0.6	2	5.11	0.002	87.35	6.57	75	3.8
		3	4.99	0.002	98.12	3.40	72	4.2
		4	4.32	0.003	139.72	12.30	68	2.8
	0.3	2	10.46	0.018	164.93	12.53	68	3.2
		3	10.03	0.011	168.11	11.97	61	3.7
		4	9.57	0.015	172.45	14.15	59	4.2

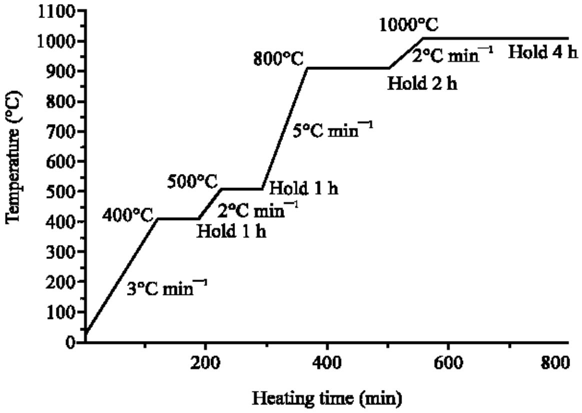

Fig. 6: The temperature rise program in the furnace

per suitable temperature rise program (Fig. 6) to produce porous aggregate with strength. In the temperature rise figure, temperature of 400°C is maintained for 1 h to heat starch and temperature of 500°C is kept to heat the ash from rice hull. The temperature of 900°C is maintained for 1 h to make kaolin convert into the metamorphic kaolin. Fu (2009) also concluded that the heating duration has its effect for sintering lightweight aggregates. But the heating rate has no significant effect. That is, the preparing and heating processes are the vital factors for manufacture of the porous aggregates.

Additive test: The black ash through simple heating and blowing treatment processes of rice husk can filter heavy metal of waste water in the patten of Wang and Lin (2009). It possesses the porous structure but do not have the strength. According to the experimental results, Chen (2008) concluded that the utilization of oyster shells, rice husk ash, WTPS and BOF slag as cement raw materials to produce cement is feasible. The oyster shell is similar to the additive of the starch in this paper. And, the sludge (WTPS) is similar to the additive of the kaolin in this paper. But the porous structure can be held for the starch and the strength can be more enhancements for kaolin without great loss of surface area. In addition, their supplies can be enough with uniform quality for their commercial characteristics. It reveals that additives possess the feasibility to improve the compressive strength and enhance porous rate in the same time while raising sintering temperature.

Table 2: Significance analysis for sintering conditions of formula (K:S:R=1:0.3:2 at 1000°C with less oxygen) by statistical method (SPSS software)

	Starch (S)		Rice hull ash (R)	
Property	F verification	Significance	F verification	Significance
Surface area	43.988	0.011	39.289	0.09
Strength	21.562	0.001	37.784	0.001
Porosity	8.757	0.121	15.256	0.161

Surface area (BET analysis), strength and porosity of different formulas in sintering at 1000°C are shown in Table 1 and 2 and we can obtain:

Effect of kaolin addition: According to Table 1 and 2, the addition of kaolin has significant impacts on the strength without great loss of the surface area. Increase of kaolin addition amount can increase the strength up to almost two times, because the kaolin assist the intense of inherent SiO_2 cristobalite faces of rice hull at sintering temperature 1000°C, so strength is the strongest. Meanwhile, its additive effect holds the surface area and postpones destroy of the porous structure, discussed above. The evidences for this trend are more obvious in Table 3. The similar results for preparing the diatomite to predict its beer filtration efficiency were concluded by Martinovic *et al.* (2006). It is also reported that the industrial waste containing SiC as the additive to raw material with appropriate treatment condition has the effect to causing the strength enhancement, density reduction and grain size holding in De Gennaro *et al.* (2007) paper. The characteristic of the additive kaolin is intensifier of the porous aggregates in this study.

Effect of starch addition: As shown in Table 1 and 2, the addition amount of starch has significant impacts on surface area holding as its standard deviation is lowing by

Table 3: Summary of the property and test analysis for different formulas and treatment conditions

				BET							
				Surface area		Strength		Water flow loss		Porosity	
Temperature (°C)	Kaolin weight ratio	Starch weight ratio	Rice hull ash weight ratio	m^2 g^{-1}	SD	psia	SD	%	SD	%	SD
900	1.00	0.3	2	75.75	3.76	142.67	8.46	0.235	0.005	67	2.5
	0.90	0.3	2	105.88	4.31	123.95	6.16	1.122	0.003	69	2.1
	0.80	0.3	2	133.41	2.92	103.47	7.95	0.475	0.004	68	3.2
	0.75	0.3	2	166.51	2.84	89.53	6.38	0.937	0.005	67	4.5
1000	1.00	0.3	2	10.46	3.43	164.93	4.98	0.223	0.002	68	3.2
	0.90	0.3	2	14.10	3.95	145.63	5.67	0.927	0.001	70	3.5
	0.80	0.3	2	16.57	4.12	140.61	8.45	0.356	0.006	65	1.5
	0.75	0.3	2	19.65	3.95	135.65	6.81	0.913	0.005	71	3.9

Table 4: Significance analysis for optimal condition (K: S: R=0.9:0.3:2 at 900°C with less oxygen) by statistical method (SPSS software)

	Temperature (T)		Starch (S)		Kaolin (K)	
Property	F verification	Significance	F verification	Significance	F verification	Significance
Surface area	28.147	0.037	30.177	0.011	32.998	0.019
Strength	48.344	0.001	31.431	0.015	37.445	0.023
water flow loss	34.953	0.008	2.519	0.041	3.156	0.049
Porosity	43.118	0.021	1.132	0.023	1.699	0.031

the addition of starch. The increase of starch addition amount can hold the surface area even a litter increase, but reduce strength, because pores increase after addition of starch, as shown in Table 3. Its adhesive effect can also lower the sintering temperature (1000-900°C) for keep the strength (164,93 psia to 142.67 psia) of the made product, also shown in Table 3. The characteristic of this additive (starch) is binder of the porous aggregates in this study. Tsai *et al.* (2006) investigate that the aggregates become lighter with high proportion of SiO_2 added to sewage sludge ash at the sintering temperature 1060. Their results also show the Al_2O_3 added to the ash raised the compression resistance and lowered the sintering temperature required. The characteristics of the adhesive effect for such additive (Al_2O_3 and starch) were also proven.

Selection of optimal formula: According to the preliminary results in Table 2, K: S: R = 1:0.3:2 is the appropriate conditions for making porous aggregate. From the product property, surface area is 10.46±0.02 mg^{-2}, strength is 164.93±12.53 psig, porosity is 68%. As for surface area of 10.46, it will be affected by water flow. Thus, this study adjusts the formula in consideration of loss on water flow. The results indicate that K:S:R =1:0.3:2 in less-oxygen state can obtain better strength and surface area, as well as the lowest loss on water flow (<0.3%). However, the formula requires more kaolin (43.48 wt. %), and increases cost. As a result, the product property is controlled by kaolin, over-high sintering temperature (1000°C) causes undesirable surface area. Thus, attempt is made to reduce kaolin amount and sintering temperature (increase surface area and reduce energy) to obtain more suitable formula.

Based on the principle of reducing kaolin and sintering temperature, the kaolin ratio is gradually reduced by 0.9, 0.8 and 0.75. The sintering temperature is reduced in each time. As shown in Fig. 7 and 8, the dosage of kaolin has no significant impact on surface area and strength, while sintering temperature has significant impact (the surface area at 1000°C is reduced to 1/5 as compared to that at 900°C). As shown in Fig. 9, the less dosage of kaolin is, the more loss on water flow is. The largest difference is about 3 times. The water treatment absorption is carried out for surface area. Fu (2009) reported that the optimal conditions of preparing lightweight aggregates from reservoir sediments are sintering temperature 1200-1250°C and duration 30 min. The properties of prepared aggregates are bulk density 1.45-1.77 g cm^{-3} and water absorbing less than 1%. Based on the water absorbing less than 1%, among all the tests, under the strength of 44.48 psia and water erosion of 90 min, the loss on water flow should not exceed 1.5% and this is the criterion for finished products' strength and loss on the water flow. The formula with optimal surface area is found based on this criterion. The results showed that 8 groups of formula meet requirements, as shown in Table 3 and 4.

As seen in the above figures and Table 3, after the heat treatment at 900°C, the finished products consisting of K:S:R = 0.9:0.3:2, meet strength requirement (123.95 psia), loss on water flow is less than 1.5% (1.1277%) and surface area is rather large (105.88 mg^{-2}). The signification value of all property is below 0.05 in Table 4. It is the evidence for the strong obvious correlation for statically SPSS analysis. The signification value of porosity is lower in Table 4 than the one in Table 3 also proves that this is the optimal formula for making porous aggregate in this

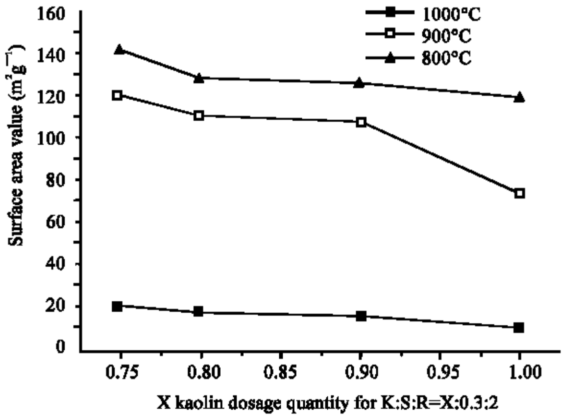

Fig. 7: The impact of surface area value with sintering temperature for dosage of kaolin

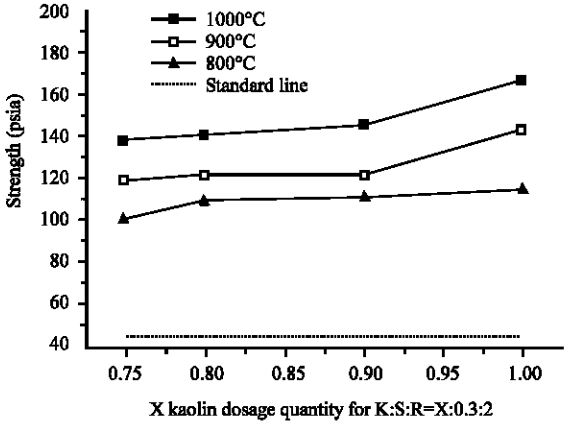

Fig. 8: The impact of strength intensity with sintering temperature for dosage of kaolin

study. Chang *et al.* (2010) reported that F-class fly ash can enhance the compressive strength, in contrast, the diatomite can keep the original porous structure. By SEM and MIP measurements, the porous rate decreases to 62.34% while sintering temperature is 1200-1270°C. The alternative quantity of fly ash increases to 20%, the compressive strength is enhanced by two times, but, the porous rate decreases to 52.94% lower. Compared with this paper, the porous rate is 67 % and the sintering temperature is 900°C in Table 3 for the optimal formula (K:S: R=0.9:0.3:2). In Table 1, the strength is enhanced by almost two times with the porous rate is keeping around 60% as the increase of the ash. That is the optimal formula and the appropriate treatment condition for making porous aggregate in this study possesses its useful and commercial application trends.

The appropriate treatment condition: The main component of the formula is an intensifier namely kaolin,

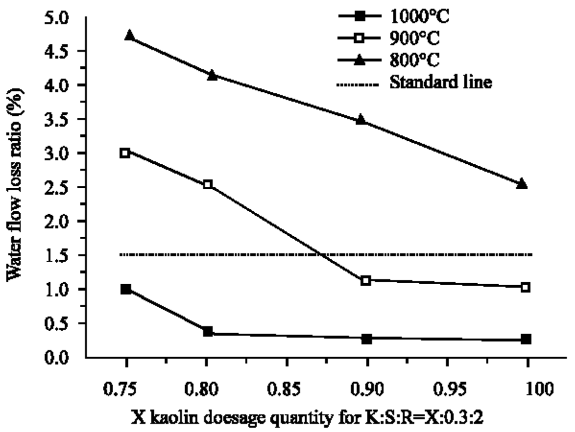

Fig. 9: The impact of water flow loss with sintering temperature for dosage of kaolin

and starch as binder mixed with ash from rice husk. A little polycyclic acid salt (5%$CaCO_3$ solution in this study) can make the black ash uniformly mix with the additive, see Leu and Lo (2007), then, through extrusion molding technique and sintering by each heating duration step in Fig. 6, the porous aggregates are produced. Besides finding a suitable formula, this processes in this study also conducted to improve the product property. The decomposition of $CaCO_3$ to release CO_2 can form the pore in the aggregates and enhance the surface area. As the analysis of the product property, the surface area (BET value) is about four times enhancement (4.65>1.51 $m^2 g^{-1}$) in some tests. The developed technique includes both the optimal formula after the appropriate treatment condition.

CONCLUSION

The additives can postpone the form of pure SiO_2 cristobalite faces with raising the sintering temperature. The main component of the formula is an intensifier namely kaolin, and starch as binder mixed with ash from rice husk.

Besides finding a suitable formula, the processes in this study also conducted to improve the product property. A little polycyclic acid salt (5% $CaCO_3$ solution in this study) can make the black ash uniformly mix with the additive and enhance the surface area. Through extrusion molding technique and sintering by each heating duration step, the porous aggregates are produced. It is evidenced the heat treatment effect is postponed by the less-oxygen state due to lean-air incineration.

Among the formulas, the formula of K: S: R = 0.9:0.3:2 at treatment temperature 900°C with less oxygen is most suitable for making porous aggregate in this study. The surface area is 105.88 $m^2 g^{-1}$, strength is 123.95psia, loss on water flow is 1.1277% and porous rate is 67%. As contrast with commercial product, the optimal formula and the appropriate treatment condition for making porous aggregate in this study possesses its useful and commercial application trends.

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