The Effects of Particle Size and Content of Rice Husk Ash from Textile Wastewater Treatment on the Properties of Portland Cement-Base Solidification

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***Abstract*—** Wastes from textile wastewater treatment consist of heavy metals that are toxic to the environment. Solidification is an alternative method to manage these pollutants. The objective of this research is to study the properties of solidification using Ordinary Portland Cement (OPC) type I mixed with rice husk ash (RHA) obtained from wastewater treatment unit owned by local textile entrepreneurs in central and northeastearn parts of Thailand. The effects of composition ratio and particle size of RHA in solidification on physical and chemical properties of mortar were investigated. Two sets of experiments were conducted by replacing cement in mortar by RHA at 20% by weight and uRHA (used RHA) at 10, 20 and 30% by weight. Three different milling times represented as three particle size distributions of RHA were studied.

***Keywords*—** Solidification, Textile wastewater, Rice husk ash, Particle size.

I. INTRODUCTION

ONE of the most popular textile wastewater treatment processes for OTOP scale entrepreneurs in Thailand is a chemical coagulation followed by physical adsorption using rice husk ash (RHA) to absorb remained pollutants from wastewater. This process generates a huge amount of used rice husk ash (uRHA). There are many disposal methods to eliminate these wastes such as burying in a landfill or burning in an incinerator. However, before burying in a landfill, these toxic wastes should be stabilized and fixed by solidification process.

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Solidification of cement-based process can be used as a waste treatment by capsulation the toxic pollutants in a form of solidified cement. This process is based on a hydration reaction or pozzolanic reaction between Portland cement and pozzolanic materials such as fly ash, cement kiln dust and rice husk ash. The pozzolanic materials must consist of siliceous or alumino-siliceous materials in finely divided form. In the presence of moisture, siliceous or alumino-siliceous chemically reacts with calcium hydroxide (formed by the hydration of Portland cement) to form compounds possessing cementing properties. Some researchers had studied the feasibility of using RHA for a pozzolanic materials due to RHA has a high silica content and high porosity. However good Pozzolanic activity of RHA must have small particle size (< 10μm) and high amorphous SiO_2 content (80 – 90% by weight). The ASTM C 618 define that the pozzolanic materials class N and F should have SiO_2+Al_2O_3+Fe_2O_3 more than 70%. The properties of cement-based solidification, such as compressive strength, leachability, depend on composition ratio and particle size of Portland cement and pozzolanic materials [1-8].

The aim of this study is to investigate the effects of RHA particle size and composition ratio on compressive strength and morphology of Portland cement-based solidification.

II. MATERIALS AND METHODS

The RHA was used as pozzolanic materials in the experiments. The fresh RHA was procured from Pathumthani, Thailand and directly used in the first part of this research without any purification process. Prior to conduct the experiments, the RHA was dried in an oven at 100ºC for 24 hours. Effect of particle size distribution was studied using three set of milled RHA at different milling time, 30, 60 or 90 minutes. Fine sands and Ordinary Portland Cement (OPC), type I was use in the experiments. The cement compositions using fresh RHA are shown as in Table I. 20% RHA was used throughout the research with different milling time of RHA from 30 (R20 +30), 60 (R20+60), and 90 (R20+90) minutes as compared with the control sample (only OPC) and unmilled RHA. The milling condition was fixed by using 120 g of RHA in 1-liter plastic bottle at a rotational speed of 210 rpm using 800 g of 8 mm ZrO_2-balls.

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The separated experiments were carried out using uRHA which was the same batch of RHA as mentioned in the previous part but the uRHA was already deteriorated after textile wastewater treatment process, collected from dyeing company in Nakornrachasima, Thailand. Effect of uRHA contents in the solidification cement was studied as we aim to fix hazardous waste into the solidification blocks. The uRHA was adjusted from 10% (U10), 20% (U20) and 30% (U30) showing in Table I.

The chemical compositions of as-received cement (OPC), RHA and uRHA were investigated by using X-ray fluorescence spectrometer (PAnalytical PW2404), while phase composition of the starting materials were investigated using X-ray diffractometer (PAnalytical X'Pert PRO) using CuK_α as irradiation source. Mixing procedure was referred ASTM C 305, while a compressive strength test was followed ASTM C 109 with three replicates of 5-cm sample cubes after 28 days curing. The leaching test of heavy metals of the U20 sample was determined using a standard test method of waste extractive test (WET) [9].

TABLE I
MIXING PROPORTIONS

Signature	Cement %	uRHA %	RHA %
Control	100		
R20+0	80		20
R20+30*	80		20
R20+60*	80		20
R20+90*	80		20
U10	90	10	
U20	80	20	
U30	70	30	

water/binder ratio = 0.75, binder/sand = 1:2.75
*Remark: 30, 60 and 90 was milled time for 30, 60 and 90 minutes

III. Results and Discussion

A. Chemical and Phase Compositions

TABLE II
CHEMICAL COMPOSITIONS OF CEMENT, RICE HUSK ASH (RHA) AND USED RICE HUSK ASH (URHA)

	Cement	RHA	uRHA
SiO_2	16.17	93.88	94.48
Al_2O_3	3.83	0.48	0.44
Fe_2O_3	3.57	0.49	0.29
Cr_2O_3		0	0.01
CuO		0	0.01
ZnO		0.01	0.01
CaO	69.45		
P_2O_5		1.02	0.89
SO_3		0.14	0.37
Cl		0.05	0.15
K_2O	0.51	1.99	1.62
CaO		1.17	1.05
TiO_2		0.04	0.03
MnO		0.15	0.2
MgO	2.24	0.48	0.31
Na_2O	0.21	0.11	0.15

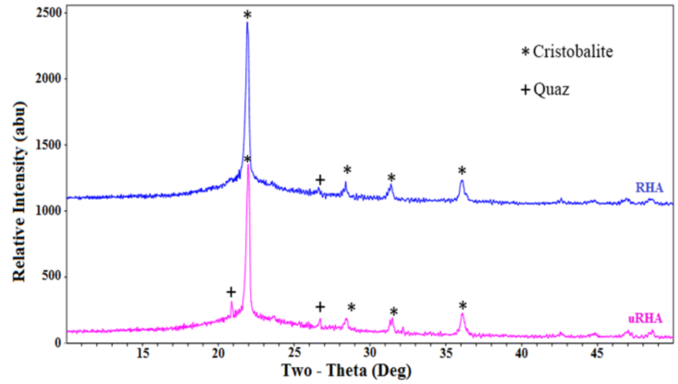

Fig. 1 X-ray diffraction patterns of RHA and uRHA

Table II shows the chemical compositions of RHA and uRHA by using X-ray fluorescence. The composition of $SiO_2+Al_2O_3+Fe_2O_3$ in RHA and uRHA were 94.85 % and 95.21 %, respectively that more than 70% corresponded to the standard of the pozzolanic materials in ASTM C618 [6].

Fig. 1 shows X-ray diffraction patterns of RHA and uRHA. It was observed that the main phase of RHA and uRHA were cristobalite, while uRHA showed second phase of quartz that might be from find sand during the filtration in the wastewater treatment process. The amorphous phase in both RHA and uRHA could be observed as the hump under the XRD patterns. There was report that an amorphous phase in pozzolanic materials could be a good reactive form for hydration reaction [3].

B. The Effect of Particle Size in Cement-Based Solidification

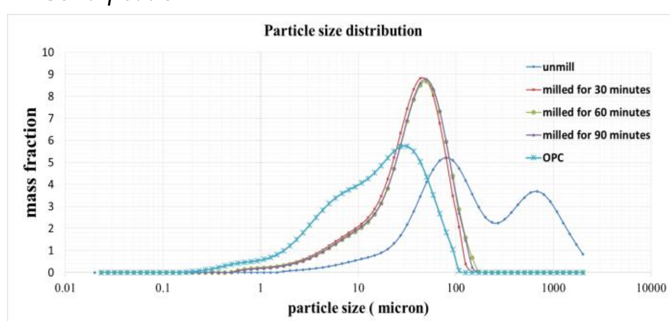

Fig. 2 Particle size distribution of RHA unmilled , milled for 30, 60 or 90 minutes and OPC

The average particle size of unmilled RHA is 273 μm and the average particle sizes of milled RHA at 30, 60 and 90 minutes were 36.26, 40.02 and 39.46 μm. The particle size of milled RHA in each condition was not smaller than 10 μm that mean there did not have good pozzolanic activity [6]. Fig. 2 shows the particle size distribution of unmilled and milled RHAs. It can be seen that unmilled RHA showed a bimodal distribution, while milled samples presented only monomodal distribution which were almost similar each other. From the results, as 30 minutes gave the same particles size distributions as obtained in 60 and 90 minutes, therefore, 30 minutes should be the optimum milling time for reducing particle size of RHA.

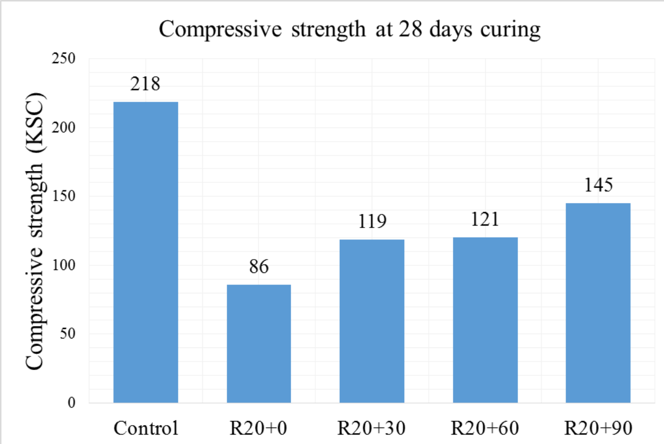

Fig. 3 Compressive strength of RHA unmilled, milled for 30, 60 or 90 minutes 20% w/w after 28 days curing

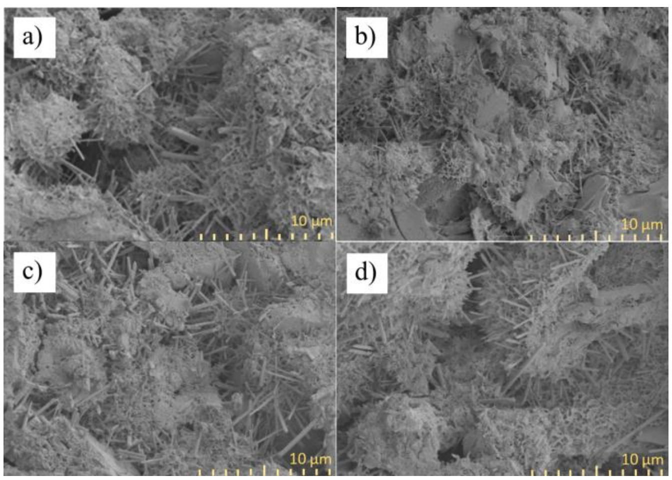

Fig. 4 SEM of cement paste of a) R20+0 b) R20+30 c) R20+60 and d) R20+90

After making cement samples using different milling time RHA at 20% content, the compressive strengths were measured and reported in Fig.3. It can be observed that the compressive strengths upon using milled RHA were much better than that the ones used unmilled RHA (the lowest improvement was 38%). However, RHA milling times (30-90 minutes) could not be clearly observed that it affected to the compressive strength of the cement samples. Only at 90 minutes of RHA milling time, the compressive strengths were significantly improved as compared with 30 and 60 minutes of RHA milling times. During cement block fabrication, the flow test of each composition was measured. We found that flow ability of the cement paste increased as the RHA milling time increased especially in 90 minutes milling time. This could be the reason why compressive strength of R+90 showed a better result. In addition, the compressive strengths of R20+0, R20+30, R20+60 and R20+90 were all higher than that the requirement of the compressive strength in the Thailand industrial standard for non-loading masonry [11]. In the other words, just 30 minutes of milling it can obtain the same particle size at 60 or 90 minutes and RHA milled for 30 minutes was enough to give compressive strength for using in the applications. Fig. 4 shows the SEM micrographs of R20+0, R20+30, R20+60 and R20+90. It can be observed the phase of ettringite, needle-like shape in all samples and the Calcium silicate hydrate (CSH) scattered with the ball shape as shown in background of SEM images. The size of ettringite in each sample was found not much different in the range of about 2 – 3 µm length and 0.5 µm diameter.

C. The Effect of uRHA contents in Cement-Based Solidification.

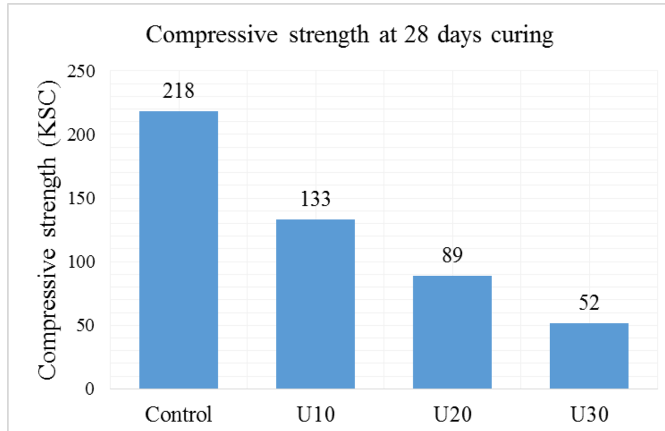

Fig. 5 Compressive strength of uRHA at 10, 20 and 30 % w/w after 28 days curing

TABLE III
LEACHING TEST OF U20

Element	STLC* mg/l	U20 mg/l
Zn	250	0.80
Cu	25	0.10
Cr	5	0.036

*Remark: Notification of Ministry of Industry, Thailand 2005

The compressive strengths of all control and uRHA 10, 20 and 30% w/w were analyzed 28 days after curing. Fig. 5 shows the change in compressive strength as a function of added uRHA. The compressive strength of sample U10, U20 and U30 was decreased about at 20, 40 and 60% of control sample (OPC only). A decrease in compressive strength as an increase amount of uRHA substitution in cement-based solidification showed detrimental effect on the hydration reaction for the development of compressive strength. However, the strengths of all samples (in u-series) were more than the minimum strength required for landfill (3.5 KSC) as per EPA guidelines [10]. In addition, an increase in percentage of added uRHA gives the flow ability to decrease. The U20 sample was chosen to measure the leachability because its compressive strength and flow ability were acceptable. The leach of heavy metal in U20 as shown in Table III was less than the standard of soluble threshold limit concentration (STLC) of Thailand [9]. Therefore the substitute of uRHA 20% is not the hazardous waste and it can utilize as cement brick application. This cement-based solidification can use for non-loading masonry materials because the compressive strength was more than the requirement of Thailand industrial standard for non-loading masonry (25 KSC) [11].

IV. Conclusions

The cement-based solidification of OPC mixed with RHA and uRHA were investigated. The following conclusion can be drawn up based on this experimental study.

1. The chemical compositions of RHA and uRHA can be used as pozzolanic materials because they contain sufficient amount of SiO_2, Al_2O_3 and Fe_2O_3 with regard to ASTM C618.
2. The compressive strengths of RHA and uRHA motar were higher than the standard for landfill of EPA guideline
3. The optimum times for milled RHA was 30 minutes as the compressive strengths of solidification cement samples obtained from milled RHA at 30, 60 and 90 minutes were almost similar, while 30 minutes milling time consumed less energy.
4. There was a feasibility for using RHA and uRHA as pozzolanic materials in construction materials because the compressive strength of RHA and uRHA mixed with OPC was higher than Thailand industrial standard for non-loading masonry.
5. Detailed of the effects of particle size on uRHA needed to be carried out as the next step of research.

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