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EFFECT OF PRETREATMENT SOAKING DURATION TO CHARACTERISTIC OF ULTRAFINE TREATED RICE HUSK ASH (UFTRHA) AS SUPPLEMENTARY CEMENTING MATERIAL (SCM)

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ABSTRACT

Rice husk ash (RHA) possesses high content of amorphous silica that is essential in pozzolanic reaction of supplementary cementing material (SCM) in concrete strength enhancement. However, usage of conventional RHA is unfavourable in current concrete industry. One of the reason that hindered the utilization of conventional RHA nowadays is due to its properties inconsistency namely chemical and mineralogical properties. In this regard, improvement on the RHA properties by introduction of chemical pretreatment prior to burning procedure is seen as an excellent way to reach the goal. In this research, raw rice husk was soaked in 0.1M hydrochloric acid (HCl) solution from one to three hours. After that, the pretreated rice husk was ground using planetary ball mill for 15 minutes. The effect of pretreatment soaking time to alkali metal removal and its chemical composition, mineralogical properties, particle size analysis as well as specific surface area of ultrafine treated rice husk ash (UFTRHA) were determined accordingly. As for alkali metal removal analysis, potassium element had the highest removal concentration with value of 55.35 mg/l. Meanwhile, amorphous silica content of the optimum sample was recorded as 96.00% at three hours of pretreatment soaking period. In terms of particle size and specific surface, it was also observed that, three hours of pretreatment soaking duration was sufficient to produce finest size of UFTRHA where d(0.1), d(0.5) and d(0.9) were obtained as 1.417µm, 4.493µm and 14.884µm respectively and the largest specific surface area of 196.61m2/g.

Keywords: treated rice husk ash, supplementary cementing material, andalkali metal removal.

INTRODUCTION

Green technology approach is essential in reducing global carbon footprint to mother earth. This phenomenon encourages explorations of the application of supplementary cementing materials (SCM) in recent concrete industry. SCM are commonly used in concrete as admixtures to enhance its properties and also to promote greener concrete production. In light of incorporating SCM into concrete production, rice husk ash (RHA) is regarded as one of them due to high silica content. Production of RHA is done via thermal activation, which is burning process of the rice husk from agricultural waste.

Rice plant is a type of plant that has the ability to take the silica content from soil. The absorbed silica is then eventually assimilates into its structure during the growth [1]. Therefore, the outer part which refers as rice husk possesses high silica content with value of more than 80% [2]. Approximately 1000 Million tons of rice is annually produced in the world, which usually leaves about 400 Million tons of rice husks as a waste material [3]. Hence, the potential of producing ash from husk is about 100 million tons per annum. Therefore, utilizing RHA in concrete is possible. Furthermore, high content of amorphous silica and high specific surface area (SSA) of RHA are possible to be achieved by adopting suitable burning condition [4]. Thus, incorporation of RHA in concrete production promotes environmental as well as economic relief. Comparing to other CRM type such as silica fume (SF) and fly ash (FA), RHA is proven to have greater reaction with portlandite ($Ca(OH)_2$) in pozzolanic reaction, contributes towards increment in compressive strength value and durability of high performance concrete [5-7] and ultra-high performance concrete as well [8-11]

A thorough study has been done on addition of conventional RHA in concrete. This is simply due to its content that possesses high amount of silica content. Nevertheless, quality of rice husk ash produced is still compromised. According to International Rice Research Institute, the rice husk is very sensitive towards incineration or burning process. As the consequences, properties consistency of conventional RHA produced is compromised. This is the main reason that leads towards unfavorable usage of this material as SCM in current construction sector [4-5]. In order to overcome the weakness of the conventional RHA properties, some of literatures have introduced chemical pretreatment procedure by using inorganic acid i.e. hydrochloric acid (HCl) and nitric acid (HNO_3) prior to burning process [5-6, 12-13]. The process was intended to remove unwanted impurities on the surface of the rice husk prior to incineration process for RHA production as SCM. In present study, the main focus is to optimize the chemical treatment parameters namely concentration of the reagent used and soaking time precisely.

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METHODOLOGY

Ultrafine treated rice husk ash (UFTRHA) preparation procedure

Raw rice husk was obtained from local rice factory (BERNAS) located in Sungai Ranggam, Perak. Prior to incineration procedure, the rice husk was treated with 0.1M hydrochloric acid (HCl) solution. The specimens were soaked in the acid solution for 1 to 3 hours. After completion of pretreatment process, the samples were washed using distilled water until it neutral pH obtained and dried using laboratory oven at 110°C. It was then burned by using muffle furnace in laboratory. The burning process was done at 700°C for 1 hour. The treated rice husk ash was then ground using planetary ball mill for 15 minutes. Figure-1 illustrated the process flow of the pretreatment procedures in this research.

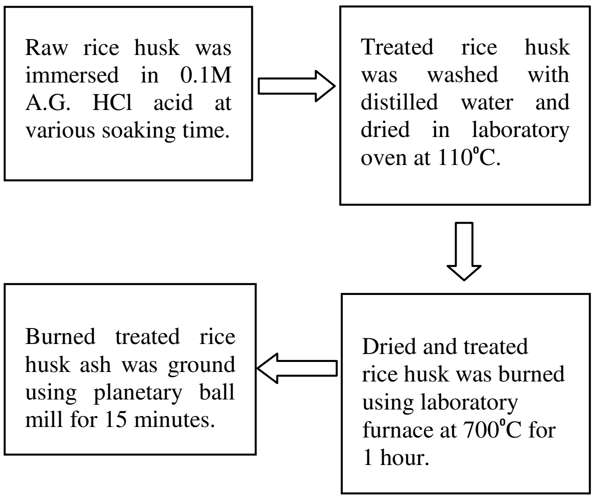

Figure-1. Process flow chart of ultrafine treated rice husk ash (UFTRHA) sample preparation.

Characterization of UFTRHA and alkali metal removal test

Effect of acid pretreatment prior to burning process of rice husk were determined via several tests. Hence, chemical composition of both ground non-treated rice husk ash (NTRHA) and ultrafine treated rice husk ash (UFTRHA) were examined using X-ray fluorescence (XRF) test. As of UFTRHA, the test was done for sample that undergone pretreatment in 0.1M HCl solution from one to three hours soaking period. The analysis was done by using spectrometer of BrukerAxs S4 Pioneer. The test was in accordance to BS EN 12677. As for mineralogical properties determination, X-Ray diffraction (XRD) analysis was done accordingly.

Brunnet-emmet-teller (BET) nitrogen adsorption test was done to examine particle specific surface area of the ground UFTRHA powder. The test was completed using surface area and pore analyzer model micromeritics ASAP 2020. Meanwhile, particle size analysiswas done using Malvern instrument mastersizer, model Scirocco 2000.

In addition, content of alkali metal impurities in the supernatant liquid from the acid pretreatment procedure was done using flame atomic absorption spectrophotometer (AAS) instrument. The leachate samples were collected from one to three hours soaking duration respectively.

RESULTS AND DISCUSSIONS

Alkali metal removal analysis

In order to measure the amount of undesirable alkali metal that has been leached out from the surface of raw rice husk, a test has been conducted using flame atomic absorption spectroscopy (AAS) equipment. For completion of this analysis, supernatant of liquid from the pretreatment process was collected and tested accordingly. Figure-2 illustrates amount of alkali metal in terms of concentration that has been removed via pretreatment procedure at various soaking period.

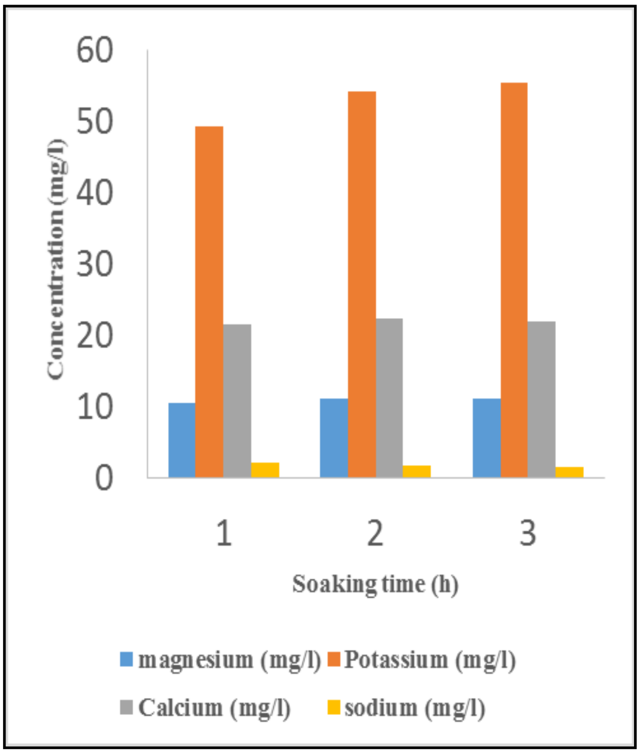

Figure-2. Alkali metal concentration leached out after pretreatment process at various soaking time.

Figure-2 shows that potassium component has highest concentration of removal followed by calcium, magnesium and lastly sodium. Removal of these alkali metal resulted from the pretreatment process is essential in order to avoid sensitivity of rice husk towards burning temperature in order to produce excellent quality of rice husk ash. According to Chandrasekhar et. al [14], potassium oxide (K_2O) is major contributor towards dark colour, in which grey to black when it melts in incineration stage. Hence, removal of potassium is in need to enhance the properties of TRHA produced.

Moreover, several researchers reported that there is a great interaction between oxides, which are potassium

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and sodium elements that presence on the surface of raw rice husk. These two components result in surface melting of silica particles and hence increase the rate of early crystallization of amorphous silica into cristobalite [15-18]. Therefore, it is essential to remove these alkali metal component via acid pretreatment prior to burning process.

Chemical composition of UFTRHA

As for chemical composition determination of UFTRHA, X-Ray Fluorescence (XRF) data is summarized in Table 1. Based on the experimental result, it can be observed that the predominant content of rice husk ash is silica (SiO_2). In addition, the SiO_2 composition in UFTRHA is greater as compared to non-treated rice husk ash (NTRHA) at all pretreatment soaking duration with percentage increment ranging from 16.61% to 17.22%.

On the other hand, potassium oxide (K_2O) content of UFTRHA, which imparts the dark colour of rice husk ash is reduced tremendously with percentage reduction of more than 98% for all UFTRHA samples. This phenomenon leads towards white colour of UFTRHA produced after completion of burning process.

Overall, all other oxides content of UFTRHA i.e. sodium oxide (Na_2O), calcium oxides (CaO) and magnesium oxide (MgO) are greatly reduced as well. These findings are in agreement with the alkali metal removal test results where potassium, sodium, magnesium and calcium concentration are traced in the pretreatment supernatant liquid. Therefore, these unwanted alkali metalsare removed from the raw rice husk surface. Removal of these impurities resulted in higher silica (SiO_2) purity percentage yield after incineration process. Hence, amount of silica (SiO_2) percentage in UFTRHA is indeed has been positively enhanced through chemical pretreatment process.

Table-1. Chemical properties of non-treated rice husk ash (NTRHA) and ultrafinetreated rice husk ash (UFTRHA).

Chemical composition	Chemical content percentage of Non-treated rice husk ash (NTRHA) burned at 700 °C (%)	Chemical content percentage of Ultrafine treated rice husk ash (UFTRHA) with incineration temperature of 700 °C (%)		
		1 hr	2 hrs	3 hrs
SiO_2	81.90	95.50	95.60	96.00
P_2O_5	3.95	2.20	0.70	2.10
Fe_2O_3	0.33	0.64	0.70	0.52
CaO	2.23	0.60	0.48	0.53
Al_2O_3	0.17	0.15	-	-
K_2O	8.21	0.10	0.04	0.06
Na_2O	0.10	-	-	-
MgO	0.34	-	-	-
MnO	0.22	-	-	-
SO_3	0.45	-	-	-
Cl	0.45	-	-	-
ZrO_3	1.62	0.46	0.25	0.38

Minerological characterization of UFTRHA

The X-Ray diffraction analysis is used to identify degree of crystallinity and amorphousness of a material. This analysis is also utilized to determine mineral presence in the tested material. Figure-3 present the XRD patterns of UFTRHA at one and three hours of pretreatment soaking time respectively.

Based on the XRD analysis, it is observed that both samples of UFTRHA are amorphous and no high intensity peaks presence. Amorphous state of a material is regarded as an indication of silica (SiO_2) reactivity that is beneficial for pozzolanic reaction. Pozzolanic activity in concrete is defined as reaction between portlandite ($Ca(OH)_2$) and reactive silica to form calcium silicate hydrate (C-S-H) that contributes towards concrete strength development. Hence, amorphous state of silica content is vital in order to produce high quality supplementary cementing material (SCM) in concrete industry application.

Physical properties of UFTRHA

Size of UFTRHA powder particle is possible to be reduced by introduction of mechanical milling mechanism using planetary ball mill. Table 2 tabulates the particle size analysis of UFTRHA at different pretreatment soaking duration from one to three hours respectively. In addition, Figure-4 illustrates the graph particle size distribution of UFTRHA at three hours soaking time and incinerated at 700 °C.

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Based on the data in Table-2, UFTRHA that had been treated for three hours has finest particle size compared to other UFTRHA samples with d(0.1), d(0.5) and d(0.9) of 1.417µm, 4.493µm and 14.884µm respectively. Meanwhile, the median value for particle size of the UFTRHA optimum sample was 4.493µm as per plotted the graph shown in Figure-4.

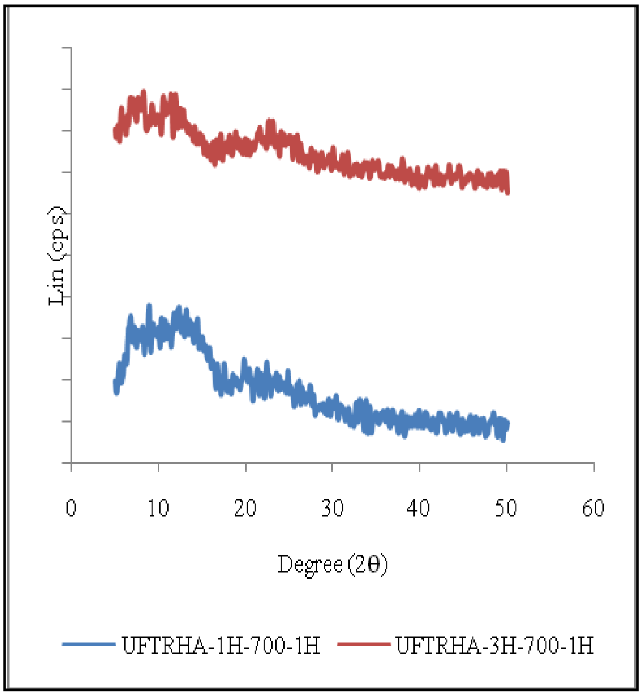

Figure-3. XRD patterns of UFTRHA at one and three hours of soaking duration.

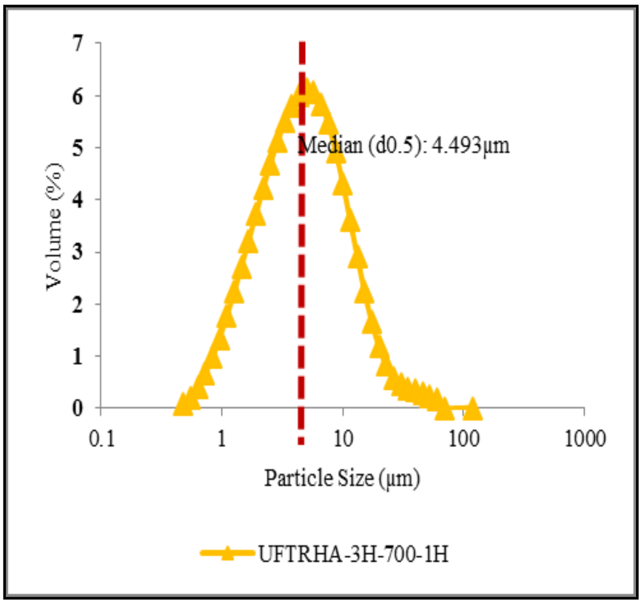

Figure-4. Median value of particle size distribution of UFTRHA at 3 hours of pretreatment soaking time and incinerated at 700 ^{o}C for 1 hour.

Table-2. Particle size analysis of UFTRHA at various pretreatment soaking time.

Pretreatment soaking time (hour(s))	Particle size (µm)		
	d(0.1)	d(0.5)	d(0.9)
1	1.434	4.552	13.996
2	1.436	4.508	14.334
3	1.417	4.493	14.884

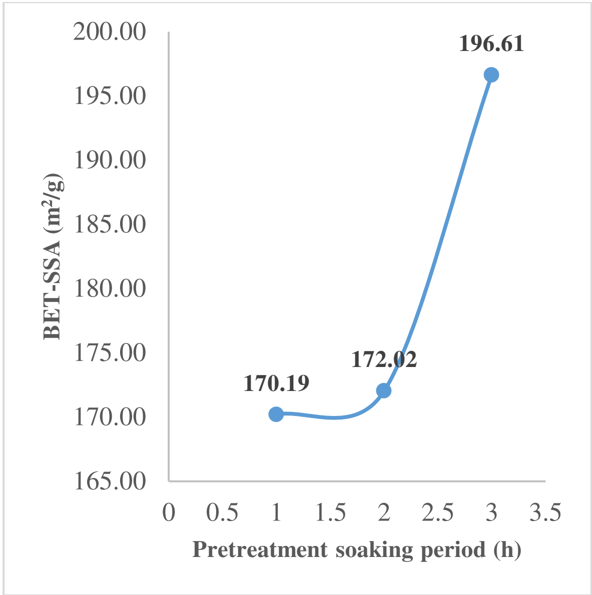

Figure-5. Effect of pretreatment soaking time using 0.1M HCl towards brunnet emmet teller specific surface area (BET-SSA) of UFTRHA.

In a nutshell, the particle size analysis and specific surface area determination for physical characterization of UFTRHA shown that soaking period at three hours using 0.1M HCl is the desirable condition to extract UFTRHA.

CONCLUSIONS

The results obtained shows that, soaking time in the pretreatment process plays an important role in terms of enhancing the chemical, physical and mineralogical properties of UFTRHA. These elements are essential in order to produce high quality supplementary cementing material (SCM) for concrete technology application. As for the chemical composition analysis, the acid pretreatment regime indeed enhanced the amount of pure silica extracted from UFTRHA from 16.61% to 17.22% compared to the non-treated sample. On the other hand, the mineralogical assessment concluded that the material, mainly silica (SiO_2) is amourphous as no sharp peak presence in XRD analysis.From this research, it can also be concluded that three hours of soaking time in pretreatment process to produce UFTRHA is the optimum period. Amorphous silica content (SiO_2) of the UFTRHA sample was recorded at 96.00% with particle size median of 4.493µm and specific surface area of 196.61 m^2/g.

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