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Effect of Incineration Temperatures and Time on the Rice Husk Ash (RHA) Silica Structure: A comparative study to the literature with experimental work

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Abstract. Controlled burning of rice husk can produce amorphous rice husk ash (RHA) with high silica content which can significantly enhance the properties of concrete. This study has been undertaken to investigate the relationship between the incineration temperatures and time to produce RHA with ultimate reactivity. The rice husk samples were incinerated in an electrical muffle furnace at 350°C, 400°C, 425°C 450°C, 475°C, and 500°C for 60 and 90 minutes, respectively. The silica structure in the Rice Husk Ash (RHA) was determined using X-Ray diffraction analysis. The results show that RHA appeared to be the totally amorphous when the husk incineration up to 425°C for 60 and even at 90 minutes. However, with increased temperature to 450°C, 475°C and 500°C, traces of crystalline silica (quartz) were detected. Nonetheless, is unable to be considered as it does not impact the ash structure. In conclusion, the result gives an idea of the temperature and the time required for the production of ash from rice husk with totally amorphous form.

Key words: Rice Husk Ash; Burning temperature; electric muffle furnace; X-Ray Fluorescence; X-Ray diffraction; pozzolanic reactivity; crystalline silica.

1. Introduction

One of the important criteria of supplementary cementing material (SCM) in concrete is to possess high content of reactive silica, as this is essential for concrete strength development. Indeed, it is proven that rice husk ash (RHA) contains high amount of amorphous silica and therefore can be used as SCM in concrete. However, producing a reactive material depends on the incineration temperature and time. Nonetheless, published research on the conversion temperature from amorphous materials contains numerous inconsistencies and contradictions (i.e. reactive) to crystalline (i.e. non-reactive) silica (Table 1).

James and Rao (1986) concluded that the transformation temperature of silica from amorphous to crystalline is related to the existence of minor elements in the rice husk ash, such as Na, K, Mg, and Ca leading to decrease of the silica transformation temperature. Therefore, the focus in this paper is to identify the impact of different incineration temperatures on the silica form of rice husk ash and to identify the ideal temperature in relation to time to generate highly active RHA.

2. Methods

The starting point of transforming rice husk ash silica from amorphous to crystalline was investigated by many researchers (see Table 1). Some studies also report on the combined effect of temperature and exposure time on the onset of silica crystallization in rice husk ash (see Table 1).

Table 1. Silica conversion temperatures in RHA from previous publications

References	Temperature (°C)	Time	Comments and results
Chandrasekhar et al. (2003)	400	6-12 h	Reactive RHA; un-burnt cellulose
	500	8-12 h	Reactive RHA; maximum reactivity
Al-Khalaf and Yousif (1984)	500	2h	High reactivity RHA
Mehta (1978)	500	prolonged period	Amorphous silica (oxidising conditions)
	680	<1 min	Amorphous silica (oxidising conditions)
Nair et al. (2008)	500-700	12 h	Amorphous silica
Khassaf et al. (2014)	550	2 h	Amorphous silica
Ganesan et al. (2008)	550-700	1 h	Amorphous silica
Asavapisit and Ruengrit (2005)	400-800	1 h	650°C optimum temperature for reactive RHA based on strength activity index.
Madandoust et al. (2011)	550-700	1 h	Amorphous silica
Huang et al. (2001)	700-800	-	Amorphous silica
Kannan and Ganesan (2014)	600-800	-	Amorphous silica
Kapur (1985)	300	15 h	Crystalline silica detected
Bui (2001)	350	15 h	Crystalline silica detected
Islam et al. (2025)	500–1000	1–6 h	Crystalline silica detected
Ismail and Waliuddin (1996)	400-700	24 h	Crystalline silica detected
Cook (1984)	< 600	-	Crystalline silica detected
Onojah et al. (2013)	> 500	-	Crystallisation begins
	600	-	Quartz detected
	800	-	Tridymite detected
	1470	-	Cristobalite detected
James and Rao (1986)	400-900	1-30 h	Reactivity of ash depends mainly on temperature; duration has small effect.
	500	-	High reactivity RHA
Chopra et al. (1981)	≤ 700	-	Predominantly amorphous, with increasing time crystals present
Ramezanianpour et al. (2009)	650	1.5 h	Amorphous silica
	700	0.5 h	Crystalline silica detected
Shinohara and Kohyama (2004)	450	6 h	Amorphous silica
	450	22 h	Amorphous silica
	700	6 h	Less than 5% Cristobalite
	800	6 h	Less than 5% Cristobalite
	900	6 h	10% Tridymite and 52% Cristobalite
Smith and Kamwanja (1986)	800	12h	Small proportions of crystalline silica
Yu et al. (1999)	≤ 750	-	Amorphous silica
	> 780	-	Crystalline $CaSiO_3$ (Wollastonite).
Hamad and Khattab (1981)	500-600	-	Amorphous silica
	800	-	Cristobalite detected
	1150		Cristobalite and Tridymite
Hanafi et al. (1980)	800-900	3 h	Amorphous silica
	900	-	Nucleation process for the formation of the low-form Cristobalite phase
Boateng and Skeete (1990)	550-700	-	Amorphous silica
	≥ 900	-	Silica transformed to crystalline
Yeoh et al. (1979)	900	< 1h	Amorphous silica
	1000	> 5min	Crystalline silica

Shinohara and Kohyama (2004) found that crystalline silica content increased by subjecting the husk to higher temperature with constant time or increasing the time at a constant temperature. Transformation process of the amorphous stage to crystalline of pure silica has been described by several authors. Kapur (1985), Boateng and Skeete (1990) and Bronzeoak (2003) give the transformation process of pure amorphous silica as follows:

$$\underset{400-600\deg C}{amorphous} \Rightarrow \underset{153\deg C}{\beta} - \underset{\substack{\uparrow\downarrow \\ \alpha - Qtz}}{Qtz} \Rightarrow \underset{117-163\deg C}{(\beta 1+\beta 2)} - \underset{\substack{\uparrow\downarrow \\ (\alpha 1+\alpha 2)-Tri}}{Tri} \Rightarrow 1470\deg C \underset{228-800\deg C}{\beta} - \underset{\substack{\uparrow\downarrow \\ \alpha - Crist}}{Crist}$$

Gorthy and Pudukottah (1999) state that silica need a certain time to gradually convert to its crystalline forms. According to the authors the formation of Cristobalite in rice husk ash could be explained as follows:

- Si–O bonds are liberated from the long polymeric chains (Si–O–C/Si–O–Si) at elevated temperatures (below crystallization point of silica).
- When the pyrolysis temperatures are increased, more and more Si–O and C–O/C–C are released, which gradually transform to crystalline silica (Cristobalite) and crystalline carbon (graphite).

3. Materials

Rice husk from Iraq was purchased for the present study. The husk particles have an average length of 6.4 - 9.1mm and width of 1.5 - 3.08mm, as shown in Fig.1. The rice husk samples as received (It underwent no chemical treatments or washing; only organic contaminants were removed.) were burned at 350, 400, 425, 450, 475, and 500 °C, respectively, for 60 and 90 minutes at exact heating temperature. The wide-angle powder X-ray diffraction (XRD) patterns of the ash samples were taken on a PANalytical X'Pert Pro MPD, powered by a Philips PW3040/60 with Mo–Kα (λ = 0.7093 Å) radiation was used to determine the silica structure of ashes samples.

Fig.1. : Boat-like shape of the as received whole rice husk.

4. Results and Discussion

4.1. Incineration process

Generally, the ignition temperature of husk in the furnace was 340°C and based on these facts, the temperature range of incineration in this study was fixed at 350°C to 500°C for two different durations (60 and 90 minutes). The oven was set up at the desired temperature before each test. When the rice husk sample (5.0g) was placed onto the steel plate inside the muffle furnace at 350°C, it was observed that the rice husk particles immediately flashed or devolatilized, resulting in flaming combustion (long trailing orange flame) for 5-9 seconds.

The resulting char particles took another 35-50 seconds to burn out entirely into ash in the form of glowing combustion with short, slight bluish flame surrounding the char particles. In fact, the final ash product from the combustion of these raw rice husk particles were not completely burned, which is clear from the color of the ash (brown). With increasing time of combustion to 90 min, the ash turned to the black color indicating wholly burned raw materials.

Sudden exposure of raw rice husk particle, when the incineration temperature increased to 400°C, caused the alkali metal compounds of potassium and sodium oxide to melt (Tapu 2018), thus forming a surface melts mixture on the char and/or ash particles. Therefore, any unburnt carbon molecules 'trapped' in this melted mixture and were unavailable for further oxidation (Krishnarao et al., 2001). In addition, resulted ash presented a variable color indicating the variation in silica structure and residual of carbon content.

Comparing the color of ash samples incinerated at 400C for 60 min and 90 min it is clear that with increasing time the color became lighter indicating a low residual carbon content. This trend became more apparent with ash prepared at 425°C and 450°C and ash prepared at 475°C to 500°C became white. These results are in contrary to the conclusion reported by Terbendalir and Taib (2007). According to the author, because of the phenomenon of surface melting, it has never been possible to obtain totally white ash from the burning of raw rice husk particles, irrespective of how long the particles were exposed to the heat.

4.2. Silica structure

All particle samples were analyzed for their silica structures using X-Ray Diffraction (XRD) analysis at the University of Brighton Laboratories. The representative XRD patterns of the RHA samples are shown in Tables 2, respectively. Based on the NIOSH manual of analytical methods (Ashley,2015) presented in Table 2, the husk burned at 350°C for 60 minute shows a weak peak of crystalline silica.

These barely observed peaks could be assigned to Cristobalite; quartz was estimated to be negligible. This indicates that the ash remained in an amorphous form. With increasing burning time to 90 min, a single diffuse band is shown at around 26.7° with slightly increase intensity from 22.03 counts (60min) to 39.71 counts. Indicated that, the silica in RHA at 350°C/90m sustains in an amorphous state. However, a comparison in between each type of rice husk degree ash, only peaks at or near of 26.66° (quartz), 21.93° (Cristobalite) and 21.62° (Tridymite) will be take in account as the primary 2°θ diffraction patterns to determine the crystallinity of ash silica. The link between time and the XRD peak indentation was depicted in Figures 2 and 3.

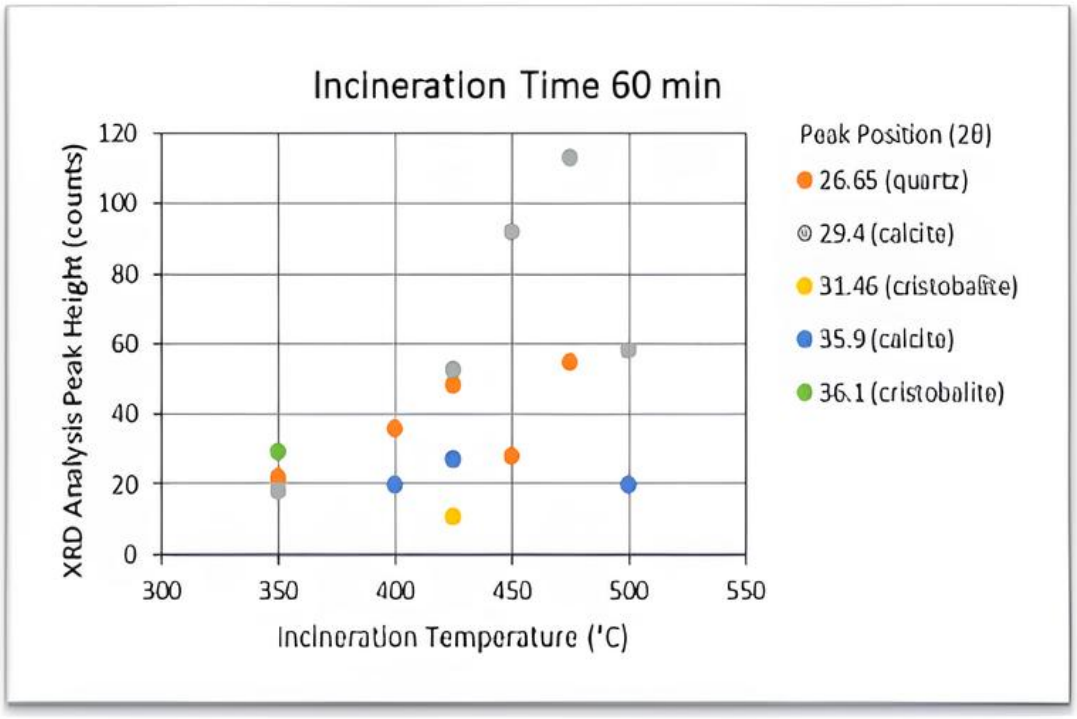


Fig. 2. Incineration temperature (60 min) to XRD peak indication.

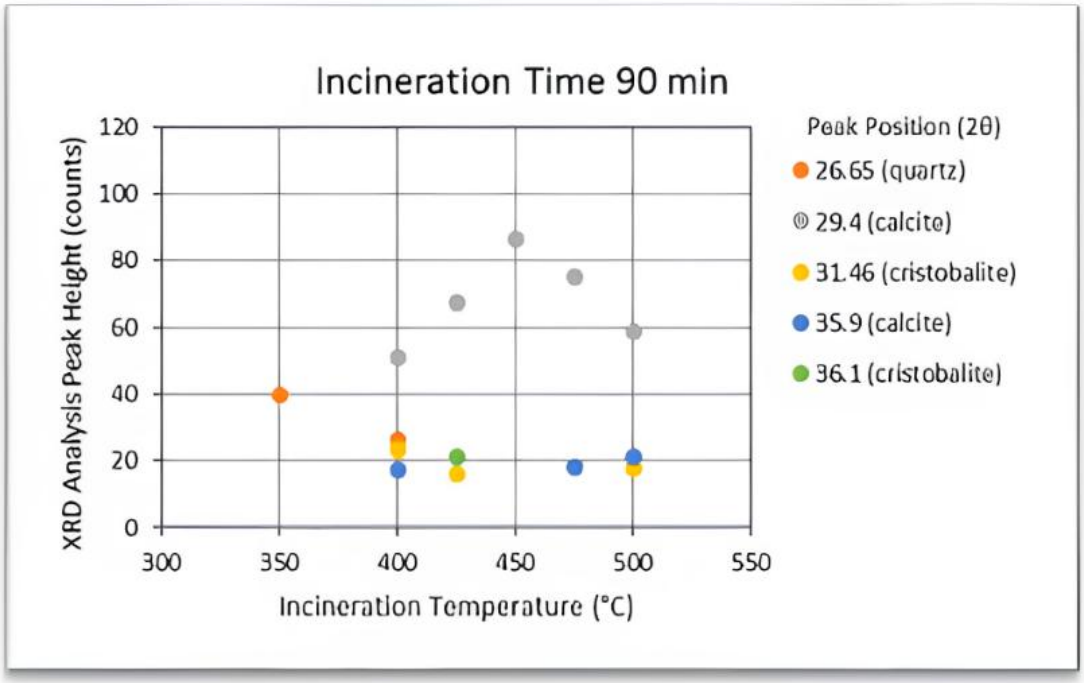
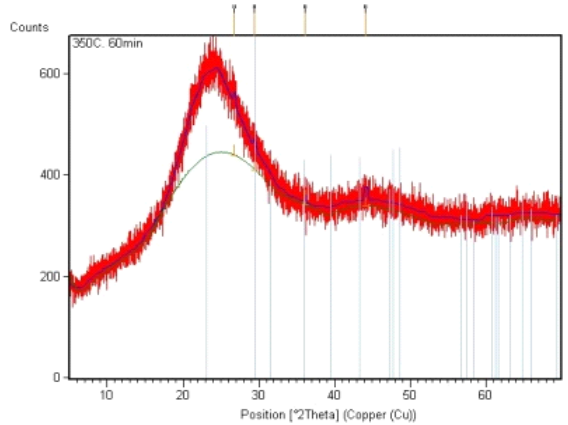
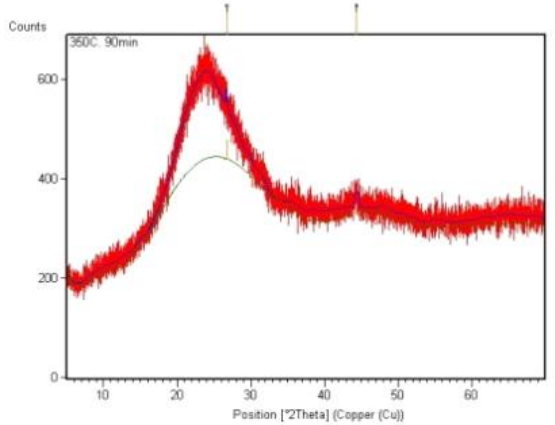
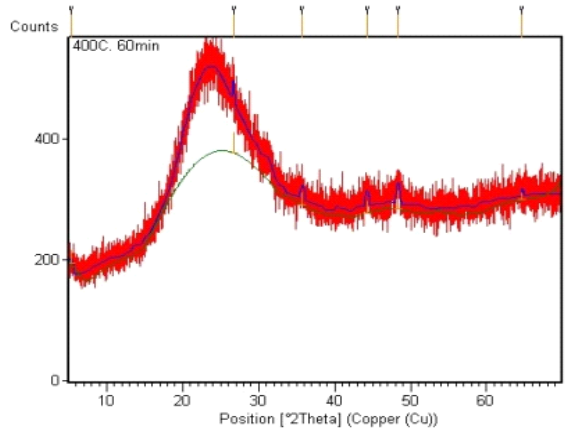
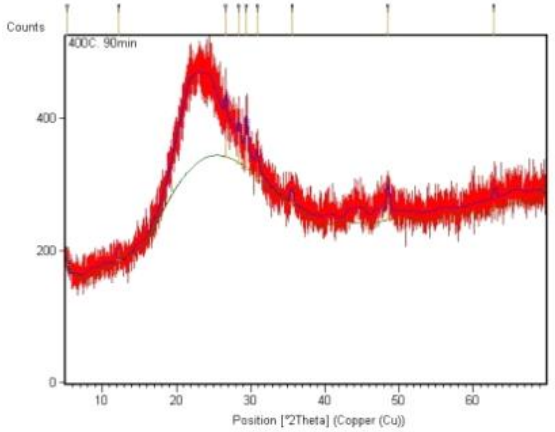
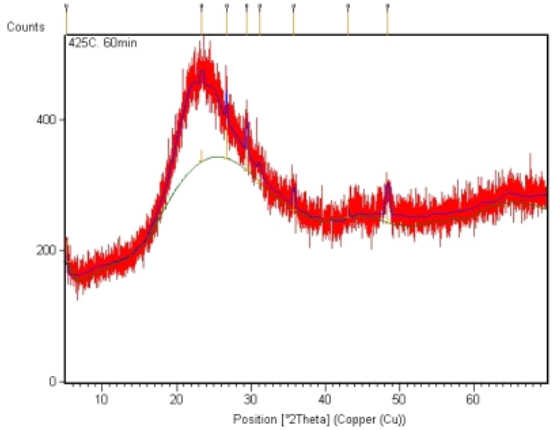
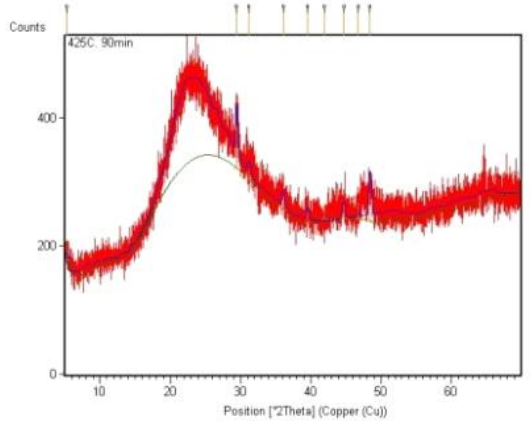
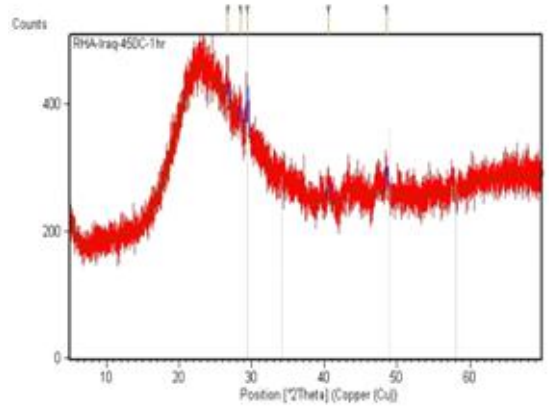
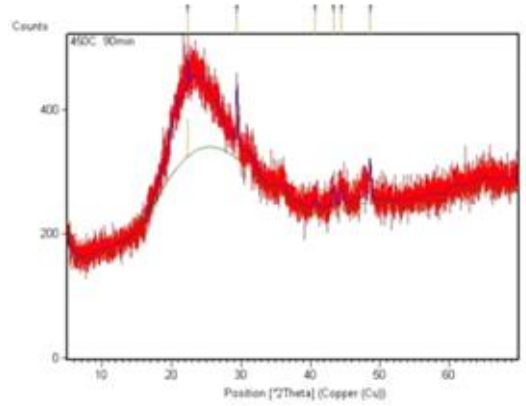
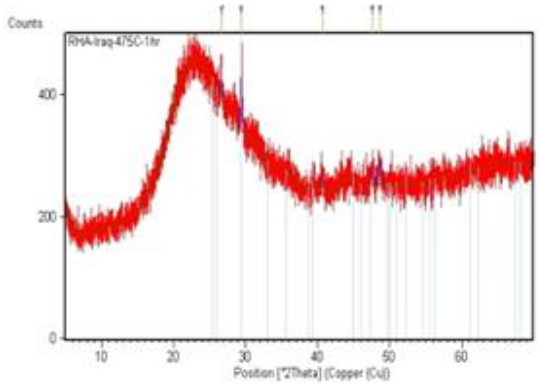
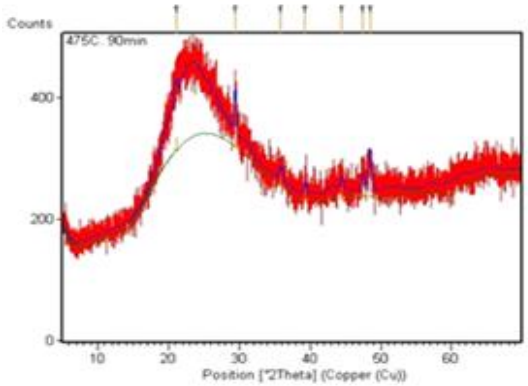
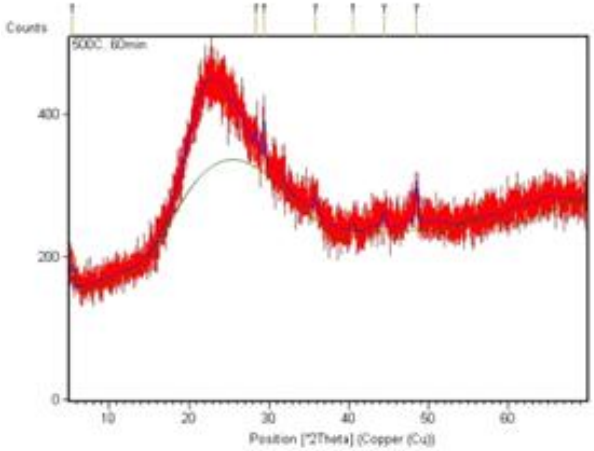
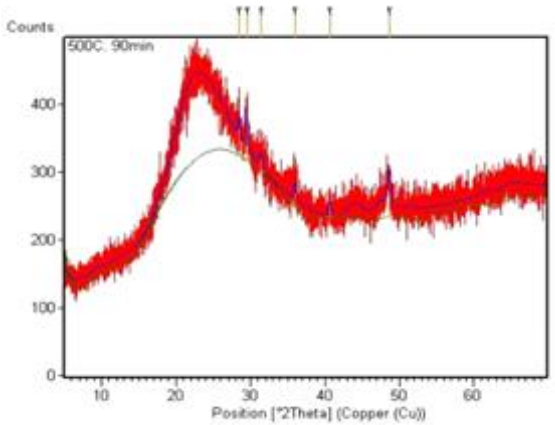


Fig. 3. Incineration temperature (time 90 min) to XRD peak indication

Table 2. Diffractograms of ash samples from the combustion of rice husk in the muffle furnace at different temperatures

Temperature [°C]	Time (min)					
	60 [min]	Peaks		90 [min]	Peaks	
		Pos. [°2θ]	Height [cts]		Pos. [°2θ]	Height [cts]
350		26.8083 29.4703 36.1190 44.1143	22.03 18.39 29.32 18.33		26.711 44.3590	39.71 21.38
400		5.2734 26.6901 35.7475 44.2214 48.3308 64.6724	22.34 35.77 20.07 21.31 32.75 12.83		5.2261 12.1744 26.6499 28.3875 29.4774 30.9161 35.5553 48.5377 62.8626	24.65 17.21 26.30 24.67 50.93 23.19 17.22 35.48 10.58

Temperature [°C]	Time (min)					
	60 [min]	Peaks		90 [min]	Peaks	
		Pos. [°2θ]	Height [cts]		Pos. [°2θ]	Height [cts]
425		5.1311 23.4072 26.7163 29.4027 31.1781 35.7709 43.1209 48.3922	32.35 19.39 48.52 52.63 10.99 27.33 26.79 31.39		5.2471 29.4707 31.2368 36.0791 39.5425 41.9505 44.6810 46.7298 48.4322	28.26 67.60 15.89 21.32 17.13 24.54 22.41 24.63 45.23
450		26.7179 28.4602 29.3922 40.5998 48.5416	28.13 28.68 92.34 28.83 32.17		22.3454 29.4680 40.6206 43.3106 44.5223 48.5886	60.70 86.32 13.16 26.17 25.58 46.94

Temperature [°C]	Time (min)					
	60 [min]	Peaks		90 [min]	Peaks	
		Pos. [°2θ]	Height [cts]		Pos. [°2θ]	Height [cts]
475		26.6578 29.4280 40.6696 47.5663 48.6096	54.78 113.18 17.55 29.57 31.81		21.0764 29.4275 35.8989 39.3276 44.5062 47.4072 48.4584	22.80 75.03 18.25 17.71 18.67 26.99 46.93
500		5.3634 28.4558 29.3829 35.8776 40.5042 44.4400 48.4534	26.97 20.20 58.62 19.82 9.42 17.18 39.11		28.4524 29.5489 31.3828 35.9354 40.6241 48.5995	34.36 58.81 17.75 21.05 18.92 33.81

5. Conclusion and Funding

Incinerating temperature degree of burning rice husk essentially control the quality of ash, which may result either in entirely amorphous form or partially crystalline. Based on this relationship according to the experimental results presented in this chapter, the following concluding remarks could be drawn:

i. The reasonably good correlation between the set of time to the burning temperature and the detected reference peaks from the XRD analysis might indicate that the degree of the ash can be transformed to crystalline silica form.

ii. An increase of burning time for each sample was not able to exhibit a noticeable increment in the intensity of the peaks of XRD. Therefore, it is believed that effect of incineration temperature might be responsible for producing less crystalline silica rather than the duration.

iii. In spite of low combustion degree (350°C), a trace of quartz can be detected; however, it doesn't affect on the ash structure.

iv. The appearance of crystallization traces peaks was highly dependent on impurity content of the rice husk. In order word, the higher the impurities, the higher the intensity of peaks.

v. Rice husk ash produced at 350°C, for 90 minutes has altogether black color, indicating of incomplete husk combustion resulting in high amount of residual carbon content.

vi. Exposure of rice husk to temperature up to 500°C for 90 minutes was sufficient to cause crystallization of silica; however, too little to make an effect therefore ignored.

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