Optimization of nanostructured/nano sized rice husk ash preparation

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Abstract

The objective of the study is developing a procedure for production and characterization of rice husk ash (RHA). The effects of rice husk (RH) amount, burning/cooling conditions combined with stirring on producing of RHA with amorphous silica, highest SiO_2, lowest loss on ignition (LOI), uniform particle shape distribution and nano structured size have been studied. It is concluded that the best amount is 20 g RH in 125 ml evaporating dish Porcelain with burning for 2 h at temperature 700 °C combined with cooling three times during burning to produce RHA with amorphous silica, SiO_2 90.78% and LOI 1.73%. On the other hand, cooling and stirring times affect the variation of nano structured size and particle shape distribution. However, no crystalline phases were found in RHA in all cases. Results proved that the Attritor ball mill was more suitable than vibration disk mill for pulverizing nano structured RHA with 50% of particle size (D_{50}) lower than 45 μm and 99 % of particle size (D_{99}) lower than 144 μm to nanosized RHA with D_{50} lower than 36 nm and D_{99} lower than 57 nm by grinding time 8.16 min to every 1 g RHA without changes in morphousity of silica.

Key words: Grinding, Nano Structured, Nano Sized, Optimization, Rice Husk Ash

Introduction

According to food and agriculture organization of the United Nations (FAO), more than 769 Mt of rice paddy are annually produced in the world (around 510 Mt, milled basis) in 2018 (1). Rice is considered as one of the main sources of food for people. Disposal of agricultural residue RH by useful way is aimed as it accounts around 20 % by weight of rice (2).

Variation in chemical composition of RH is found from one sample to another due to the differences in the climate and geographical conditions (3). But, generally, RH is composed of both organic and inorganic matter. The organic element analysis shows that the average organic composition of rice husk was 39.8-41.1 wt.% carbon, 0.5-0.6 wt.% oxygen, 5.7-6.1 wt.% hydrogen and 37.4-36.6 wt.% nitrogen while silica is the major component of inorganic minerals (4). However, uncontrolled combustion of RH produces RHA with low reactivity (5). The preparation of RHA should be optimized to be used as a partial replacement of Portland cement (6). The partial replacements of Portland cement depend on pozzolanic activity which depends on parameters such as content of amorphous silica, specific surface area of particles and particle size distribution (7).

According to (8-11), production of amorphous silica with some carbon and metallic impurities obtained from burning of RH depends on rate of heating, ultimate temperature, retention time, atmosphere cooling rate, and grinding time of the ash. Temperature ranging below 800 °C and higher

than 500 °C was highly recommended. In this regard, incineration of rice husk at temperatures below 500 °C is not desirable and produces an amount of unburned carbon which can result in adverse effects on ash pozzolanic activities. On the other hand, burning at temperature higher than 800 °C produces crystalline silica instead of amorphous one, which is not desirable due to the decrease in the pozzolanic reactivity. However, in all cases quickly cooling of RHA by directly removing it from the oven is preferable to avoid crystallization. Also, grinding is recommended to increasing the pozzolanic reactivity by increasing the specific surface area and decreasing the particle size.

In this study, we focus on studying the effect of RH amount, cooling times with stirring and grinding process. Finally, the chemical and physical properties of nano structured RHA, before and after grinding, were determined, to obtain high reactive and optimal RHA suitable for replacements of Portland cement according to (ASTM C618) (12). The effect of both nanostructured and nanosized produced RHA was studied in terms of physical and mechanical properties of cement and mortar (13).

Materials and methods

The sample of RH used in the study was obtained from Kafr El Sheikh, Delta, Egypt. Its chemical composition is shown in Table 1.

Table 1.Chemical composition of RH as received

Chemical composition	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Na_2O	K_2O	LOI	Sum
(%)	16.33	0.22	0.08	1.04	0.02	0.03	0.03	0.36	81.78	99.88

The sample preparation procedures involved were:

1. Sorting of RH to confirm that there is not any contamination with other materials during handling, transportation and storing.
2. Soaking the RH for 24 h in distilled water to dissolve any dissolved impurities.
3. Washing of 5 Kg RH using of 40 L distilled water several times with shaking.
4. Drying of RH was done in a room temperature 25 °C and humidity 20 % for 2 days.
5. Optimization of firing time and RH amount (20, 30 and 100 g) to obtain RHA.
6. Burning 20 g RH in 125 ml evaporating dish Porcelain for 2 h at temperature 700 °C.
7. Cooling one time at the end of the burning for 2 h up to 3 times of cooling for 5 min up to 6 times of cooling rate to every 20 min of burning process.
8. Optimization of grinding time for RHA to the nano scale which particle sizes smaller than 100 nm was studied by using vibration disk mill (HERZOG, Model HSM 100 H, Grinding vessel chromium steel 100 cm^3 and continuously variable rotation speed 750 – 1500 rpm). The mass ratio of grinding media to RHA powder was 30:1. And the particle size was determined every 2 h by using Zeta-sizer Nano ZS and checked by using TEM. Further grinding using Attritor ball mill (model, Union Process, Model# 1S) has been applied. The controlled parameters of the Attritor ball mill, which were held

constant, were mass ratio of ball to RHA powder 8:1, milling rotational speed 1170 rpm. Particle size distribution D_{50} and D_{99} was being analyzed every 6 h of grinding time by using BT-9300S Laser Particle Size Analyzer.

9. Loss on ignition was measured according to (EN 196/2) (14) using muffle furnace, (model, Eurothengleby VECSTAR).
10. The chemical analysis was measured according to (EN196 – 2.2:2005) (15) and (ASTM C114) (16) by using X-ray fluorescence apparatus, (Model ARL 9900 by Thermo Scientific).
11. Selection of the best method to determine RHA particle size depends on using of Zeta-sizer Nano ZS (Malvern Instruments, Worcestershire, UK) then particle size distribution D_{50} and D_{99} at different grinding time was measured by using Laser diffraction apparatus combined with laser diffraction-SSA, laser particle size analyzer (Model BT-9300S by Bettersize) according to the International Standard ISO 13320 (17). The particle size of RHA before grinding was tested by using sieving test according to (ASTM C618) (12) to avoid irregular shapes and to confirm the fineness requirements for fly ash and natural pozzolana to be less than 34 % of the material retained on the 45 μm sieve. Also, the particle size of final grinding time sample was confirmed by using TEM, (Model JEOL JEM-2100).

12.   Specific surface area was measured by using electronic air permeability apparatus ToniPERM for Blaine (Model3301/204/06 Zwick/RoellbyToni Technik) according to (EN196-6) (18) and (ASTM C204) (19) and by using Brunauer-Emmett-Teller (BET) calculation according to ISO 9277 (20) by using nitrogen adsorption method, (Model TriStar II by Micromeritics) and pore size distribution by using BJH method.

13.   Mineralogy was measured by using XRD, (X – Ray diffractometer, Panalytical Embryean, Model No.: 202964).

Results and discussion
Chemical analysis

The chemical analysis in Table 2 shows that the SiO_2 content of 100 g RH was affected slightly (only 3 %) by increasing burning time from 1 to 2 h. On the other hand, it increased 25.5 % by decreasing RH amount from 100 g to 30 g and increased 29.4 % in case of decreasing RH amount to 20 g.

Table 2. LOI and Chemical analysis of RH and RHA at different burning time and amount

Chemical compostion (%)	RH after washing and drying	100 g RH after 1 h burning	After burning for 2 h			
			100 g RH	30 g RH	20 g RH	20 g RH as received
SiO_2	15.18	57.33	60.33	85.82	89.76	85.62
Al_2O_3	0.20	0.81	0.80	1.08	0.98	1.45
Fe_2O_3	0.42	0.44	0.31	0.45	0.40	0.70
CaO	0.81	3.28	3.50	4.89	4.67	6.62
MgO	0.01	0.20	0.22	0.47	0.57	0.60
SO_3	0.02	0.00	0.00	0.12	0.13	0.54
Na_2O	0.00	0.00	0.00	0.09	0.00	0.00
K_2O	0.15	0.49	0.53	0.85	0.77	1.70
LOI	83.14	37.27	34.16	6.15	2.28	2.37
Sum	99.93	99.83	99.85	99.92	99.56	99.60

Fig.1 shows that the highest SiO_2 and lowest LOI contents are achieved by decreasing the amount of RH. Comparing the results of burning RH as received and after pretreatment, the washing of RH increased SiO_2 content by 4.1%. This is due to dissolved impurities released by washing such elements as dust and dissolved calcium and potassium component. These impurities cause decreasing CaO and K_2O content by 2% and 1%, respectively after burning which reflected directly on SiO_2 increasing.

The results of LOI tests presented in Table 2 indicate that LOI of RHA is more affected by RH amount than burning time,
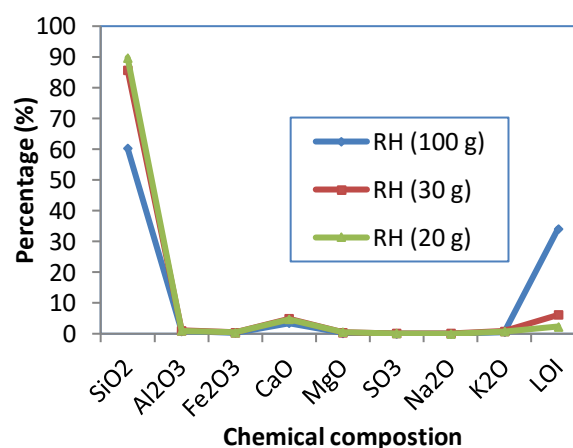

Figure1. The effect of RH amount on chemical composition and LOI

as the burning time increased from 1 h to 2 h, LOI slightly decreased from 37.27 % to 34.16 % compared to high decreasing from 34.16 % to 6.15 % caused by RH amount decreased from 100 g to 30 g, then dropped to 2.28 % when RHA decreased to 20 g. This is attributed to decreasing RH amount resulting in more specific surface area facing heat by decreasing the evaporating dish capacity. Also, LOI is a factor of quantity. It was found that the lowest LOI in 2 h is achieved when the amount of RH (g)/evaporating dish volume (ml) is equal to 0.16. On another side, LOI didn't affect by RH washing process.

Effect of cooling

The cooling times increased SiO_2 content due to the renewing of oxidizing conditions to remove more carbon. It was observed (Table 3) that when the number of cooling periods increased from one to three up to six times, LOI decreased from 2.28 to 1.73 to 1.3 % respectively. This may be attributed to the renewing of oxidizing conditions during opening the muffle furnace gate and extraction of crucible for cooling and stirring which to fire residual carbon.

Table 3. LOI and chemical analysis of RHA at different fast cooling periods

Chemical compostion (%)	Number of cooling periods		
	1	3	6
SiO_2	89.76	90.78	90.94
Al_2O_3	0.98	0.93	1.04
Fe_2O_3	0.40	0.42	0.37
CaO	4.67	4.34	4.48
MgO	0.57	0.58	0.61
SO_3	0.13	0.13	0.10
Na_2O	0.00	0.00	0.00
K_2O	0.77	0.74	0.80
LOI	2.28	1.73	1.30
Sum	99.56	99.65	99.63

Effect of grinding

After grinding, LOI increased from 1.73 % to 6.73 %, as shown in Table 4, when particle size decreased from D_{50} 45 μm to D_{50} 36 nm. Grinding for a long time will decrease SiO_2 by 9.6 % due to the increase of LOI by 5 % after grinding which may be attributed to the extraction of internal

carbon content remained in internal structure of RHA . The increase in Al_2O_3 may be attributed to contamination coming from the grinding mill components.

Particle size, distribution and shape of silica

The RHA which cooled three times was the best due to achieve the higher content of amorphous SiO2 by 1% increase and the lower amount of LOI than RHA cooled one time and approximately had the same analysis of RHA cooled six times. However, it has lower variation in SiO_2 size and the particle shape distribution shows more uniformity than RHA cooled one and six times as shown in Fig. 2, so this sample has been selected to apply the grinding process.
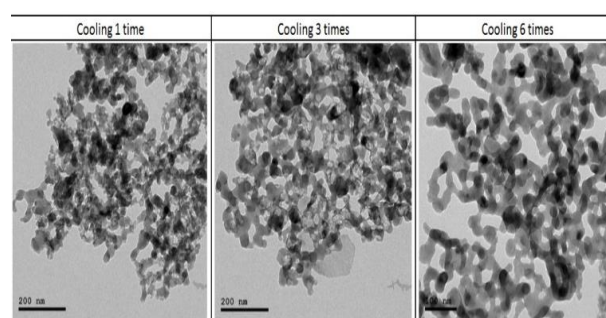

Figure 2. Effect of cooling times on TEM micrograph

Zeta-sizer was used to measure the particle size in the case of grinding by using vibration disk mill. Fig. 3 shows the dramatic decreasing of RHA particle size to 433.9 nm measured by using 0.5 % concentration of RHA with 0.5 % dispersing agent isopropanol and sonication time for 30 min for the de-agglomeration and increasing the dispersion efficiency .

The coarse RHA particles with irregular shapes and lightweight before grinding were founded. So, particle size was difficult to track by the stable way using Zeta-sizer. RHA without grinding was 3667 nm which less than the value after grinding one hour, 4000 nm. During the sieve test, RHA percentage retained on mesh (90μ) was 4.8 % and 33.2 % retained on mesh (45μ) which confirmed that it is not suitable for RHA particle size to be tracked by Zeta-sizer apparatus due to its irregular shapes and light weight. The grinding of RHA for 6 and 8 h resulted around 200 nm as shown in the TEM micrograph Fig.4. While Zeta sizer resulted in around 450 nm for the same samples. For tracking the particle size, BT-9300S Laser Particle Size Analyzer was used. It is featured by auto water level measurement and auto dispersing that ensure the accuracy. However, the resulted particle size range agrees with TEM results.

Table 4. LOI and chemical analysis of RHA before and after grinding

Chemical compostion (%)	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Na_2O	K_2O	LOI	Sum
RHA before grinding	90.78	0.93	0.42	4.34	0.58	0.13	0.00	0.74	1.73	99.65
RHA after grinding	81.21	4.94	0.65	4.61	0.47	0.10	0.00	0.87	6.73	99.58
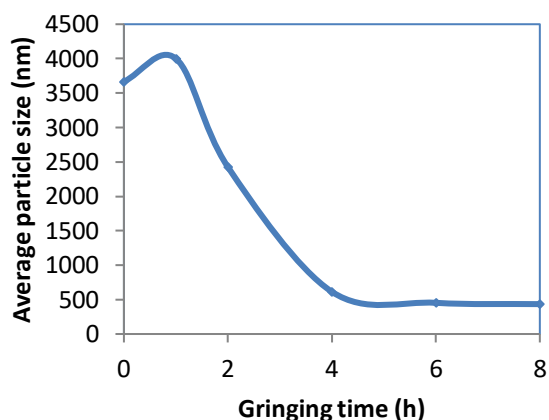
Figure 3. Effect of grinding on average particle size

Black spots appeared in TEM micrograph (Fig. 4). This is attributed to using vibration disk mill. When the vessel temperature increased to 90 °C, the RHA particles adhered to each other and form layered sheets.
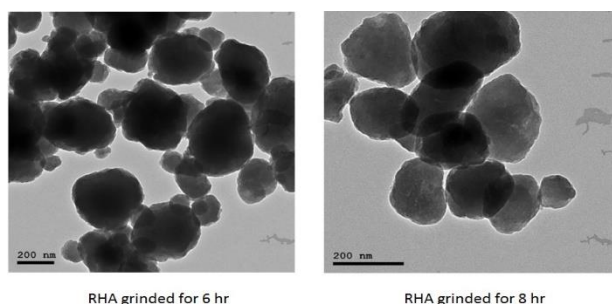

RHA grinded for 6 hr   RHA grinded for 8 hr

Figure 4.TEM micrograph for six and eight hours grinded RHA.

In order to avoid the appearance of that black spot and prevent adhesion as well as increasing productivity, Attritor ball mill combined with cooling system have been applied. . Table 5 shows the particle size distribution D_{50} and D_{99} using BT-9300S Laser Particle Size Analyzer, it was being analyzed every 6 hours grinding time by using Attritor ball mill.

Particle size distribution D_{50} and D_{99} of RHA decreased dramatically to D_{50} 7 μm and D_{99} 19 μm with increasing grinding time to 30 h (Fig.5). After 30 h of grinding, the particle size started to increase until 42 h to D_{50} 21 μm and D_{99} 36 μm then decreased dramatically to D_{50} 36 nm and D_{99} 57 nm after 102 h grinding. Decreasing rate of particle size was high during the first period of grinding within 6 h also after 48 h and slows with time above 6 h until 30 h. This may be attributed to the elongated grinding time which gains the particles extra charges that reinforced the agglomeration

Table 5. Effect of grinding time on Particle size

Time (h)	0	6	12	18	24	30	36	42	48	54	60	66	72	78	84	90	96	102
D_{50} (μm)	45	21	17	12	10	7	16	21	10	5	1	0.76	0.32	0.09	0.07	0.05	0.04	0.036
D_{99} (μm)	144	65	43	32	26	19	25	36	19	13	7	2	0.93	0.13	0.09	.08	0.06	0.057

Surface analysis

The grinding of RHA from micro to nano scale using Blaine method caused increasing of surface area from 0.2997 m²/g to 2.0849 m²/g (Table 6). However, there are some limitations for using the Blaine measurement. It does not reflect the accurate RHA specific surface area (SSA) specially when it is larger than 0.5 m²/g, so it becomes unreliable (21). Another method was tested, laser diffraction-SSA. It was found that, as grinding time increase, the SSA of RH increased slightly started from 0.0864 m²/g, after 72 h, a sudden increase happened till surface area increased to 29.4811 m²/g with increasing the grinding time to 102 h, it appears clearly in Fig.5. These results agree with BET surface area (29.794 m²/g) as stated in Table 6 which indicates that the internal surface area disappeared due to grinding effect.
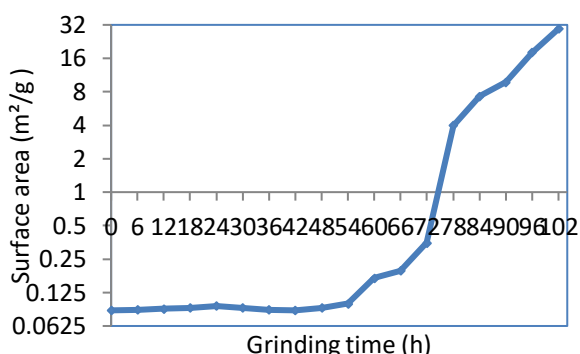
For the RHA before grinding, the BET surface area is 87.517 m²/g. After grinding, it reaches 29.794 m²/g. The significant drop in surface area occurring after grinding due to particles agglomeration occurs during the grinding process. The enormous decrease in internal surface area was confirmed by cumulative volume of pores results. The pore volume decreased from 0.127 cm³/g to 0.042 cm³/g (Table 6). This decreasing in RHA internal surface area was due to the destroying the cellular structure existing in the RHA during grinding. This conversion of RHA into tiny particles filled the spaces and decreased the volume of pores.

Most of the pore size lies in the mesopores region (2-50 Å) (Fig. 6). Using the N_2 gas adsorption/desorption isotherms (Fig.7), the hysteresis can be classified as typical mesoporous of type IV isotherm according to IUPAC classification (22).
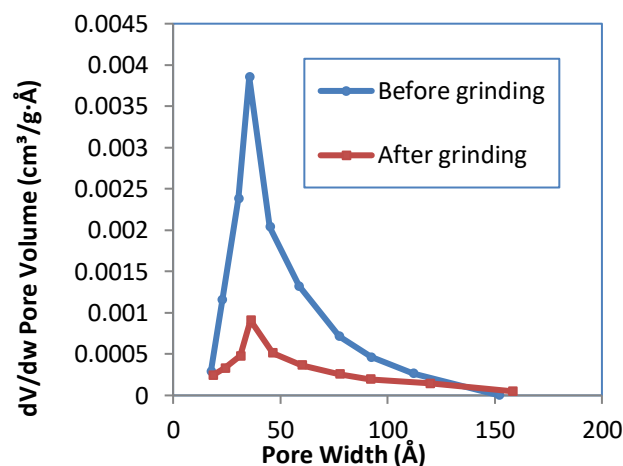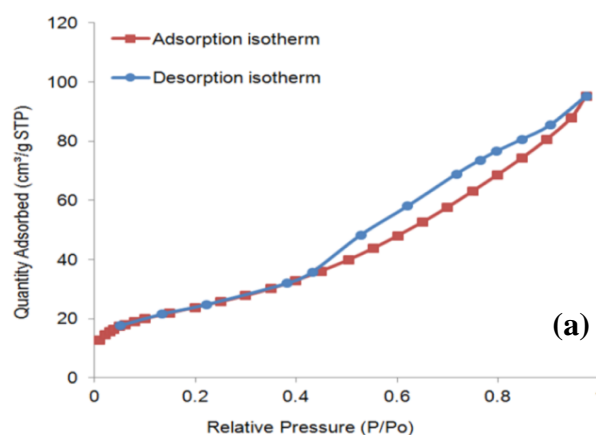

Figure5.Effect of grinding time on specific surface area of RH

Table 6.Comparison of Blaine-SSA, Laser diffraction-SSA, BET-SSA for RHA before and after grinding

Surface analysis	RHA before grinding	RHA after grinding
Blaine surface area [m²/g]	0.2997	2.0849
Laser diffraction surface area [m²/g]	0.0864	29.4811
BET surface area [m²/g]	87.517	29.794
Cumulative volume of pores [cm³/g]	0.127	0.042
Average pore width [nm]	4.321	4.972

Figure 6. **Pore size distribution of RHA before and after grinding**
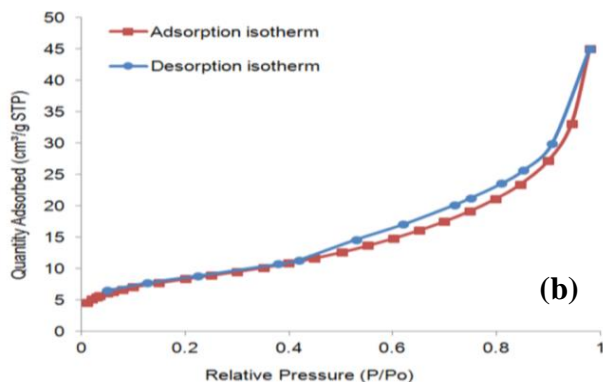
(b)

Figure 7. Adsorption/ desorption isothermals of RHA: (a) before grinding and (b) after grinding

Mineralogy

No obvious change in mineralogy is noticed between XRD diffractograms of RHA before and after grinding (Fig. 8), both of them have **(b)** broad peak of RHA with high content centered on range ≈ 22-23° (2θ) which is characteristics of amorphous SiO_2 (23). Also, XRD results confirmed that avoiding long period of burning prevents crystallization, which is observed for temperatures exceeding 700°C. Increasing the retention time produces amorphous RHA. Also, the grinding process does not affect the mineralogy of RHA.
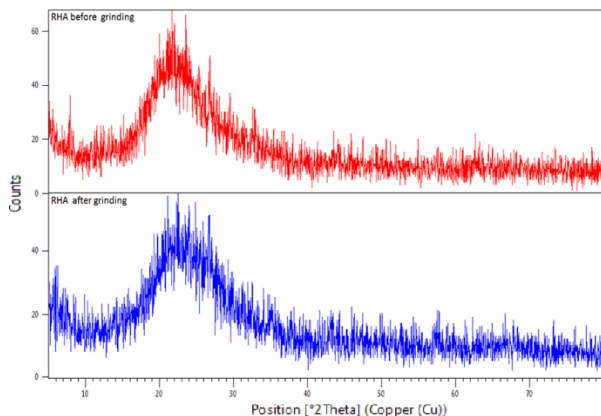

Figure8. Mineralogy state of RHA before and after grinding.

Conclusion

The optimum conditions to produce nano structured RHA with highest amorphous SiO_2 and lowest LOI was burning for 2 h at temperature 700 °C combined with cooling 3 times and mass of rice husk (g)/volume of the evaporating dish (ml) is 0.16. These optimum conditions resulted 90.78 % of amorphous SiO_2 and 1.73 % of LOI . Using Attritor ball mill is the best to convert micro RHA to nano RHA combined with uniform particle shape distribution. Tracking RHA particle size requires laser particle size analyzer featured by auto dispersing which ensures the accuracy and

overcomes the RHA irregular shapes and light weight. Tracking RHA specific surface is more accurate by using BET method due to RHA cellular structure. Grinding RHA doesn't affect its mineralogy and adsorption/ desorption isothermals type, but it decreases the BET surface area.

Authors' declaration:

- Conflicts of Interest: None.
- We hereby confirm that all the Figures and Tables in the manuscript are mine ours. Besides, the Figures and images, which are not mine ours, have been given the permission for re-publication attached with the manuscript.
- The author has signed an animal welfare statement.
- Ethical Clearance: The project was approved by the local ethical committee in Beni-Suef University, Beni-Suef, Egypt.

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تعظيم استفادة إنتاج جزيئات السيليكا النانومترية من قشر الأرز

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الخلاصة

تم تطوير إجراء لإنتاج رماد قشر الأرز . وقد تم دراسة تأثير العوامل المؤثرة على إنتاجية وخصائص السيليكا غير المتبلورة وهذه العوامل هى الكمية الابتدائية لقشر الأرز و ظروف الاحتراق و التقليب . تم التوصل إلى أن أفضل كمية كانت 20 غراما في الدقيقة في صحن تبخر خزفي بحجم 125 مل يحترق لمدة ساعتين عند درجة حرارة 700 درجة مئوية ، مع تبريد على ثلاث فترات الاحتراق لإنتاج السليكا عند أقل فقد عند الاشتعال وقد وجد أيضا أن فترات التبريد تلعب دوراً هاماً فى تباين حجم الهيكل النانوي وتوزيع شكل الجسيمات. بالاضافة إلى أنه لم يتم العثور على مراحل بلورية في رماد قشر الأرز في جميع الحالات.

الكلمات الافتتاحية: قشر الرز، الأحتراق، التبريد، جزيئات نانومترية، سيليكا، طحن