Synthesis and characterisation of submicron silica particles from rice husk

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Rice husk is considered an important silica source. In this work, microsilica particles were obtained by incineration of rice husk and milling of its ash. The synthesis process was carried out by incinerating the rice husk at 700°C, followed by acid leaching in order to remove inorganic impurities and finally mechanical ball milling at 0, 18, 36 and 72 h to reduce the particle size. Thermogravimetric analysis shows the thermal degradation of rice husk and elimination of organic components. The chemical composition was studied by X-ray fluorescence. The structural properties of silica were determined by X-ray diffraction. The morphological changes caused by mechanical milling were studied by scanning electron microscopy (SEM). The results indicate that between 150 and 450°C, the organic material of the husk was released, and above 550°C, ash with high silica content was obtained. Acid leaching increased the silica purity from 93.40 to 98.22%. According to the X-ray diffraction analysis, the predominant phase was silica. The reduction in the crystallinity of silica samples was shown as an effect of mechanical milling while the particle size was reduced. The percentage of crystallinity decreased from 33.5 to 28.5% for after 72 h of milling. The SEM images showed that the particle size reduced with an increase in milling time, obtaining particle diameters smaller than 2.0 µm. The amorphous silica obtained from rice husk can be used for the production of high-performance silicon, silicon carbide, silicon nitride, silicon tetrachloride and zeolites.

1. Introduction

Rice is a fundamental part of the human diet. According to the Food and Agriculture Organization of the UN, the global production of rice in the year 2016 was 748.0 million t.¹ As a result of rice production and refinement, each year, large amounts of husk are generated as a by-product or agricultural biomass.² Its commercial cost is insignificant, but it is considered a natural silica source, which is used as fertilizer for land,³ as fuel in heat generation due to its high calorific value (approximately 13–16 MJ/kg),⁴ for obtaining pozzolanic material⁵ and for biogas production. It can be also used as Portland cement additive^{6–8} and for the production of thermal energy, and it is considered as a source of lignocelluloses used in the preparation of bio-oil.^{9–11}

About 20% of the volume of grain rice is husk, so that around 144 million t of rice husk (RH) is produced each year.¹² In Colombia, about 793.880 metric t of RH is produced per year.¹³ However, the accumulation of biomass has increased environmental concern. Using agricultural waste instead of conventional materials offers a very important environmental contribution; it helps to reduce the problems related to pollution and conservation of natural resources.¹⁴ This large amount of husk produced can generate problems related to solid wastes, and if it is not handled properly, it would increase the risk of fire,

attract disease-carrying animals and even occupy large spaces in landfills or end up being dumped into water sources, causing release of methane (which is a greenhouse gas) during its decomposition and contaminating the environment and affecting people's health.^{2,15,16} Due to the rapid development of green technology, there is a growing tendency to produce materials from renewable sources.¹⁷ With the calcination of organic materials, thermal decomposition occurs and ash is obtained in a fine particle size; when mixed with lime, it can produce a material with binding properties, due to its pozzolanic activity.¹⁴

In the rice plant (*Oryza sativa*), silica that is stored in the husk enters in a soluble form, as a monosilicate acid, through the roots and it moves to the outer surface of the plant, where it becomes concentrated by evaporation and polymerisation and a membrane of silica and cellulose is formed.¹⁸ On the other hand, the RH contains about 75–90% organic matter (cellulose, lignin and hemicellulose), and the rest (10–25%) corresponds to other inorganic elements such as silica and traces of elements (oxides).^{12,14,19} Of these inorganic components, silica represent about 94% of its weight, while the remaining 6% corresponds to oxides as calcium oxide (CaO), potassium oxide (K₂O), magnesium oxide (MgO), aluminium oxide (Al₂O₃), phosphorus pentoxide (P₂O₅), manganese oxide (MnO) and iron (V) oxide (Fe₂O₃).¹⁵

Heat treatment is used to reduce the organic compounds in RH. Inorganic impurities or trace metals are removed by acid chemical treatment, in order to transform these impurities into soluble ions and remove them by filtration.¹⁹ Finally, a white powder rich in silica is obtained with a high surface area.²⁰ Singh *et al.*,²¹ Kalapathy *et al.*²² and Yalçin and Sevinç²³ concluded that rice husk ash (RHA) is an excellent source of highly pure amorphous silica. Amorphous silica is a porous material, with a high superficial area and several applications in polymers, electronics, catalysis, ceramics, pharmaceuticals, manufacture of dental materials, thermal insulation, rubbers, fine ceramic powders and cement additives, and it is used in the industry of photoelectric materials such as thixotropic agents and filler compounds.^{19,24–26} Nowadays, silica from RH is prepared using several methods, including sol–gel, vapour-phase reaction, thermal decomposition technique, thermal pyrolysis, chemical precipitation and biotransformation method.^{12,20} However, these techniques are complex, expensive or require a large amount of energy to transform ash into silica. For this reason, low-cost methods for obtaining microsilica from RH should be developed. The ball milling method is not used widely in the synthesis of silica particles from RH. This technique has many advantages such as low cost, effectiveness, high purity of products, moderate temperature reaction, good process control, easy operation and reproducibility.²⁷

Several studies have focused on the production of microsilica and nanosilica particles by expensive techniques such as chemical and biological methods;^{12,28,29} however, more work is required that leads to the implementation of low-cost methods. The purpose of this work was to obtain silica from RH and then convert it into microsilica particles through mechanical ball milling, which is a versatile and low-cost technique. The purity of the silica and the change in the morphology and the structural properties during the reduction of particle size in relation to the grinding time were studied. Many researchers have studied alternative uses for this waste during the last decade.²² Some research studies have shown that ash is used as a source of highly pure amorphous silica.^{23–26,30} It is also used for the production of high-performance silicon and its derivative compounds such as silicon carbide, silicon nitride, silicon tetrachloride and zeolites.¹²

2. Experimental procedure

2.1 Microsilica synthesis

2.1.1 RH incineration

RH was collected from the central zone of Colombia (Tolima). RH was washed four times with distilled water to remove impurities and other contaminants, and then it was dried in the oven at 100°C for 3 h. The RH was then burnt in a furnace; the temperature was raised at a rate of 5°C/min to 400°C and was maintained isothermally for 1 h to remove organic material. Then, the temperature was increased to 700°C and was maintained isothermally for 1 h to increase the amount of silica oxide by reducing the carbonaceous materials.²⁷

2.1.2 Elimination of inorganic impurities

For the chemical treatment, distilled water, sulfuric acid (H₂SO₄), nitric acid (HNO₃) or hydrochloric acid (HCl) was used. The acids were purchased from Colombia Bioquigen Ltd and were analytically pure. RHA was leached to remove impurities through a chemical reaction between the acid and the metallic impurities. These reacted metals are leached from the acid solution during filtration.³¹ Ash samples of 15.0 g were added to 250 ml of hydrochloric acid, nitric acid or sulfuric acid solution (1.0 M) and stirred for 1 h at 90°C. The obtained paste was washed four times with distilled water, filtered and dried in a conventional oven at 100°C for 3 h. After the leaching process, a white powder rich in silica was obtained, as can be seen in Figure 1.

2.1.3 Mechanical ball milling

A unitised jar mill model 753RM, commercialised by U. S. Stoneware, equipped with a ceramic container and 20 mm dia. ceramic cylinders as grinding media were used. A cylinders/powder ratio of 10:1 was used at a frequency of 100 revolutions per minute for the mechanical ball milling process. Samples were prepared with different milling times and compared with a control sample. Table 1 shows the experimental conditions of this process.

2.2 Methods of characterisation

2.2.1 Thermogravimetric analysis

The analysis of the thermal properties of the RH was performed using a TGA Q500 instrument (TA Instruments, USA). Samples

Figure 1. Photographs of the transformation of the ash with the acid leaching process: (a) RHA; (b) white ash with high content of silica

Table 1. Mechanical ball milling experimental conditions

Sample (ash)	Milling time: h	Label name
1	0	C0
2	18	C18
3	36	C36
4	72	C72

of 6.5 ± 0.1 mg were placed in a platinum crucible and heated at a heating rate of $10^\circ\text{C}/\text{min}$ from room temperature to 700°C . The ramps were conducted under the dynamic air atmosphere ($60\text{ ml}/\text{min}$). The maximum rates of degradation and decomposition steps were determined through the peaks defined in a derivative curve. The mass loss associated with organic material content and residue was determined using the Universal Analysis 2000 TA software.

2.2.2 X-ray fluorescence analysis

X-ray fluorescence (XRF) was used in order to determine the chemical composition of RHA. Previously, the ash samples were dried at 105°C for 12 h. Then, the powder samples were mixed with spectrometric wax (Merck & Co.); a sample/wax ratio of 10:1 was used. The slurry was homogenised by means of stirring and compacted to generate 37 mm dia. pellets for the analysis. The semi-quantitative analysis was performed with the software SemiQ performing 11 scans to detect components, excluding the lighter elements (hydrogen (H), carbon (C), lithium (Li), beryllium (Be), boron (B), nitrogen (N), oxygen (O)) and transuranic ones. A Philips MagixPro PW-2440 XRF spectrometer equipped with a rhodium tube with a maximum power of 4 kW was used. The equipment has a sensitivity of 200 parts per million (0.02%) in the detection of heavy metal elements. Table 2 shows the experimental conditions for the samples.

2.2.3 Morphological analysis by scanning electron microscopy

A Jeol JSM 5910 LV scanning electron microscope was used with 15.00 kV electron acceleration voltage, in order to study the morphology of RHA. Prior to the analysis, the samples were fixed on copper (Cu) specimens held on carbon adhesive tape. A gold layer approximately 20 nm thick was deposited by sputtering on ash surface to make it conductive.

2.2.4 Structural analysis by X-ray diffraction

Crystalline phases presented in RHA were identified using Rigaku diffractometer equipment, MiniFlex II, operated at room temperature and equipped with a copper $K\alpha$ radiation source ($\lambda =$

Table 2. Experimental conditions for the acid leaching process for the RHA samples

Sample	Conditions
1	Without leaching
2	Leached with sulfuric acid
3	Leached with nitric acid
4	Leached with hydrochloric acid

$1.540562\text{ \AA}$), and a 30 kV and 15 mA X-ray source. Measurements were run from 5 and 70° on a 2θ scale with a step size of $0.02^\circ/\text{s}$. In order to find the percentage of crystallinity, the free version of the MDI Jade 5 software was used.

3. Results and discussion

3.1 Thermogravimetric analysis

Figure 2 shows the thermogravimetric analysis (TGA) curves, showing the amount of mass remaining from the combustion of the husk as a function of temperature. The derivative profile from differential thermal thermogravimetric analysis (DTGA) gives the rate of change of mass loss as the temperature rises. The thermograph of Figure 2 identifies three characteristic events in the decomposition process. The events were delimited with the help of the peaks in the DTGA derivative. Event 1 ranges from room temperature to 140°C , event 2 between 150 and 350°C and event 3 between 350 and 500°C . In the first stage (dehydration), the curve shows an initial weight loss of 5.87%. This loss can be interpreted as the elimination of physically adsorbed water by the porosity of the husk and external water bounded by surface tension.³⁰ In the second stage, corresponding to the highest peak in DTGA, the active pyrolysis zone was identified. In this process, hemicellulose and cellulose are decomposed into small molecules and then released as gaseous volatiles. When this reaction is carried out, carbon and oxygen are removed from the polymer, and mainly carbon dioxide (CO_2) or carbon monoxide (CO) is released.³⁰ In this process, 48.05% of the mass of the sample was removed. In the third stage, the so-called passive pyrolysis zone, 20.14% of the mass was released due to the lignin combustion process.^{24,25} After 550°C , the mass remains stable and the ash (25.57%) corresponds to the inorganic residue. The analysis of residue was done with XRF (reported in the following); the RHA consists mainly of silica.²⁴

The temperature peak values represent the highest rate of change of mass for the three stages: 44.13 , 304.30 and 425.86°C , respectively. The variables measured in TGA and DTGA are

Figure 2. RH TGA and DTGA curves at air atmosphere at $10^\circ\text{C}/\text{min}$

Table 3. TGA/DTGA characterisation of the RH

Event	TGA values		DTGA		Identification
	Temperature range: °C	Weight loss: %	Maximum peak temperature: °C		
1	25–140	5.87	44.13		Moisture/water elimination
2	150–350	48.05	304.30		Hemicellulose and cellulose degradation
3	350–500	20.14	425.86		Lignin degradation

summarised in Table 3. These values are in agreement with those from the studies of Pandey *et al.*,³⁰ who reported that the three main constituents of the lignocellulosic materials (hemicellulose, cellulose and lignin) are chemically active and decompose thermochemically at a temperature range between 150 and 500°C. They also reported that the decomposition of hemicellulose starts first at a range between 150 and 350°C, followed by the decomposition of cellulose between 275 and 350°C. In addition, these two components contribute mainly to the formation of the volatile compounds, while lignin, whose decomposition occurs between 250 and 500°C, is mainly responsible for the char portion of the product.

3.2 Analysis of inorganic impurities

The chemical composition of ash was obtained by XRF for four samples with different acid leaching processes. The results are shown in Table 4. The leaching process converts the metallic elements into soluble ions; then, these are removed during filtration.⁹ According to Table 4, the ash leached with acid presents an increase in its silica content and a reduction of impurities; the acid leaching process decreased the level of these oxides. Before the chemical treatment, the silica content in the ash was 93.40%. After the treatment, the content rose to 98.22%; this indicates that the impurities represented mainly in metallic oxides were eliminated. In relation to the effectiveness of the solvent for removing impurities, it can be inferred that there are no significant differences among them.

3.3 Morphological analysis

The effect of milling time on the morphology and particle size of RHA was investigated. Figure 3 shows the micrographs of the four samples with milling times of 0, 18, 36 and 72 h at ×100, ×1000

and ×20 000 magnifications, respectively. The effect of milling time on the morphology and particle size of RHA was investigated. The scanning electron microscopy (SEM) images of Figure 3 show the effect of milling time for three magnifications. The control sample (C0) showed larger grains, even with particle agglomerates that reach approximately 10 µm. On the other hand, with the increase in milling time (from 18 to 72 h), the size of silica particles decreased: even microparticles measuring less than 3 µm can be observed. A detailed view at ×40 000 magnification of the three samples C18, C36 and C72 are shown in Figure 4. Some agglomerates of silica micron and submicron particles and the formation of cumuli among the particles can be seen. The effect of milling time on the reduction in the mean particle size is again observed. For both milling times of 18 and 36 h, the particles reach a size lower than 3 µm, while the sample with a milling time of 72 h (C72) presents a finer grain size that is even smaller than 1 µm; there are also some isolated particles in the submicron (nano) size (see Figure 5). These results agree with those of Liou *et al.*³²

A more detailed view at ×80 000 magnification of C72 is presented in Figure 5. The particles, which are presented as clusters, have a size less than 1.0 µm; this phenomenon can be associated with electrostatic attraction or van der Waals forces.³³ These results clearly show that the particle size decreased as the mechanical milling time increased; microparticles and even agglomerates of silica nanoparticles can be obtained. Increasing the milling time (from 0 to 72 h) reduces significantly the particle size in C18, C36 and C72 samples, because the system is subjected for longer to the collisions, intensive fracturing and welding generated in the milling process. Mechanical ball milling is shown to be effective for reducing the particle mean size of the silica.

Table 4. Chemical compositions of RHA with and without chemical treatment

Element and/or compound	Without chemical treatment: %	Leached with sulfuric acid: %	Leached with nitric acid: %	Leached with hydrochloric acid: %
Silicon dioxide (SiO ₂)	93.40	98.22	98.44	98.48
Calcium oxide	0.70	0.27	0.26	0.25
Potassium oxide	0.68	0.71	0.63	0.60
Phosphorus trioxide (P ₂ O ₃)	0.59	0.05	0.04	0.06
Magnesium oxide	0.26	0.21	0.22	0.20
Aluminium oxide	0.21	0.14	0.11	0.10
Manganese oxide	0.14	0.10	0.10	0.10
Iron (III) oxide (Fe ₂ O ₃)	0.09	0.10	0.09	0.08
Sulfur trioxide (SO ₃)	0.07	0.10	0.04	0.05
Chlorine (Cl)	0.06	0.06	0.04	0.05
Na ₂ O ₃	0.02	0.03	0.02	0.02
Others	3.78	0.01	0.01	0.01

Figure 3. SEM micrographs at ×100, ×1000 and ×20 000 of ash samples leached with nitric acid, for different milling times

3.4 Structural analysis by X-ray diffraction

The X-ray diffraction (XRD) patterns of the RHA for different milling times (0, 18, 36 and 72 h) are shown in Figure 6. Silica oxide was the principal phase identified through the patterns; the

arrows in the figure correspond to the localisation of main diffracted peaks of silica oxide. These results were compared with database code AMCSO 0001629 of silica oxide. The most prominent peak is at $2\theta = 22.01^\circ$. On the other hand, Figure 6

Figure 4. SEM micrographs at ×40 000 of ash samples leached with nitric acid for different milling times

Figure 5. SEM micrographs at $\times 80\,000$ of ash samples leached with nitric acid with 72 h of mechanical ball milling

also shows that peaks at high angles tend to disappear with increasing milling time. This fact is clearly noticed when comparing the patterns corresponding to 0 and 72 h of milling. The effect of milling was corroborated. For 18, 36 and 72 h of milling, the peaks corresponding to the planes (113) and (212) disappeared, and the more prominent peak (101) was widened and became less intense; other peaks ((111), (102), (200), (301)) were also attenuated, which indicates that the crystallinity of the silica was reduced as a consequence of the milling process.

In order to determine the influence of milling time on the structure, Figure 7(a) shows the peak [101] for the samples C0, C36 and C72. It is clear that the intensity decreases and the width

Figure 6. XRD patterns of RHA for different milling times

peak increases as the milling times were increased. The reduction in the intensity can be correlated with the structural changes in the silica, and an increase in stacking faults and structural disorder widens the XRD peaks. This widening is also related to the reduction in the particle size as shown in SEM images. To determine the influence of the milling time on the structure, the changes in the intensity and the full width at half maximum (FWHM) of the peak can be correlated with the structural changes in the sample. Mechanical milling always introduces stress that manifests itself with the widening of the diffraction peaks. The values corresponding to the FWHM were obtained using a Gaussian model and are shown in Figure 7(b).

The samples with 72 h of milling time showed the higher value of FWHM. Two facts can be attributed to this widening: the

Figure 7. (a) The peak [101] and its Gaussian approach of the samples at different milling times. (b) Full width at half-maximum (FWHM) curve as a function of milling time

reduction in particle size and the mechanical stress in the material. It can be interpreted as a structural distortion effect, an increase in hardness and density of point defects originated by the collisions, which affect the crystallinity, and grain boundary mobility; this fact is more evident for a higher milling time.³⁴

Finally, the crystallinity percentage values were calculated in order to determine the effect of mechanical milling. Values of 33.5, 31.9, 30.7 and 28.5% were obtained for samples C0, C18, C36 and C72, respectively. The FWHM changes showed that milling time changed the structure because the high FWHM is related to a lower crystallinity of the samples.

4. Conclusion

In this work, a versatile and efficient method for obtaining submicron silica particles from the incineration of RH and mechanical milling of its ash was developed. TGA showed that the decomposition of RH occurred in three stages corresponding to water removal, active pyrolysis and passive pyrolysis. Above 550°C, the mass stabilised and a residue was obtained. XRF analysis showed that the residue was essentially silica. The acid treatment proved to be effective for removing impurities; this process generated increases of 5% in ash purity. The predominant structural phase shown by XRD was silica. The attenuation of peaks in larger 2θ angles and a decrease in the intensity of the main peak at $2\theta = 22.01^\circ$ were observed. The crystallinity degree was reduced as a consequence of the milling process. The SEM images showed that at 72 h of milling, agglomerates of submicron silica particles with a diameter smaller than 1.0 μm were obtained. RH is considered a natural material source of silica which can be used as an additive in cement and provide added value to this abundant waste product.

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REFERENCES

- Food and Agriculture Organization of the United Nations (2016) *Seguimiento del mercado del arroz de la FAO*. Food and Agriculture Organization of the United Nations, Rome, Italy (in Spanish).
- Prasetyoko D, Ramli Z, Endud S, Hamdan H and Sulikowski B (2006) Conversion of rice husk ash to zeolite beta. *Waste Management* **26**(10): 1173–1179.
- Della VP, Kühn I and Hotza D (2002) Rice husk ash as an alternate source for active silica production. *Materials Letters* **57**(4): 818–821.
- An D, Guo Y, Zhu Y and Wang Z (2010) A green route to preparation of silica powders with rice husk ash and waste gas. *Chemical Engineering Journal* **162**(2): 509–514.
- Chindaprasirt P, Kanchanda P, Sathonsaowaphak A and Cao HT (2007) Sulfate resistance of blended cements containing fly ash and rice husk ash. *Construction and Building Materials* **21**(6): 1356–1361.
- Chindaprasirt P, Jaturapitakkul C and Rattanasak U (2009) Influence of fineness of rice husk ash and additives on the properties of lightweight aggregate. *Fuel* **88**(1): 158–162.
- Ganesan K, Rajagopal K and Thangavel K (2008) Rice husk ash blended cement: assessment of optimal level of replacement for strength and permeability properties of concrete. *Construction and Building Materials* **22**(8): 1675–1683.
- Nehdi M, Duquette J and El Damatty A (2003) Performance of rice husk ash produced using a new technology as a mineral admixture in concrete. *Cement and Concrete Research* **33**(8): 1203–1210.
- Liou T and Wu S (2010) Kinetics study and characteristics of silica nanoparticles produced from biomass-based material. *Industrial & Engineering Chemistry Research* **49**(18): 8379–8387.
- Zheng JL and Kong YP (2010) Spray combustion properties of fast pyrolysis bio-oil produced from rice husk. *Energy Conversion and Management* **51**(1): 182–188.
- Wang G, Wang D, Kuang S, Xing W and Zhuo S (2014) Hierarchical porous carbon derived from rice husk as a low-cost counter electrode of dye-sensitized solar cells. *Renewable Energy* **63**: 708–714.
- Tolba GMK, Barakat NAM, Bastawesy AM et al. (2015) Effective and highly recyclable nanosilica produced from the rice husk for effective removal of organic dyes. *Journal of Industrial and Engineering Chemistry* **29**: 134–145.
- Sierra Aguilar J (2009) *Alternativas de aprovechamiento de la cascarilla de arroz en Colombia*. MSCE thesis, Universidad de Sucre, Sincelejo, Colombia (in Spanish).
- Cobrerros C, Reyes-Araiza JL, Manzano-Ramírez A et al. (2015) Barley straw ash: pozzolanic activity and comparison with other natural and artificial pozzolans from Mexico. *BioResources* **10**(2): 3757–3774.
- Carmona VB, Oliveira RM, Silva WTL, Mattoso LHC and Marconcini JM (2013) Nanosilica from rice husk: extraction and characterization. *Industrial Crops and Products* **43**(1): 291–296.
- Shen Y, Zhao P and Shao Q (2014) Porous silica and carbon derived materials from rice husk pyrolysis char. *Microporous and Mesoporous Materials* **188**: 46–76.
- Zhang Z, He W, Zheng J, Wang G and Ji J (2016) Rice husk ash-derived silica nanofluids: synthesis and stability study. *Nanoscale Research Letters* **11**(1): 502.
- Sun L and Gong K (2001) Silicon-based materials from rice husks and their applications. *Industrial & Engineering Chemistry Research* **40**(25): 5861–5877.
- Deiana C, Granados D, Venturini R et al. (2008) Activated carbons obtained from rice husk: influence of leaching on textural parameters. *Industrial & Engineering Chemistry Research* **47**(14): 4754–4757.
- Zhang H, Zhao X, Ding X et al. (2010) A study on the consecutive preparation of D-xylose and pure superfine silica from rice husk. *Bioresource Technology* **101**(4): 1263–1267.
- Singh D, Kumar R, Kumar A and Rai KN (2008) Synthesis and characterization of rice husk silica, silica-carbon composite and H_3PO_4 activated silica. *Cerâmica* **54**(330): 203–212.
- Kalapathy U, Proctor A and Shultz J (2003) Silicate thermal insulation material from rice hull ash. *Industrial & Engineering Chemistry Research* **42**(1): 46–49.
- Yalçın N and Sevinç V (2001) Studies on silica obtained from rice husk. *Ceramics International* **27**(2): 219–224.
- Kamath SR and Proctor A (1998) Silica gel from rice hull ash: preparation and characterization. *Cereal Chemistry* **75**(4): 484–487.
- Liou TH (2004) Evolution of chemistry and morphology during the carbonization and combustion of rice husk. *Carbon* **42**(4): 785–794.
- Pijarn N, Jaroenworarluck A, Sunsaneeyametha W and Stevens R (2010) Synthesis and characterization of nanosized-silica gels formed under controlled conditions. *Powder Technology* **203**(3): 462–468.

27. Liou TH (2004) Preparation and characterization of nano-structured silica from rice husk. *Materials Science and Engineering: A* **364**(1–2): 313–323.
28. Salavati-Niasari M, Javidi J and Dadkhah M (2013) Ball milling synthesis of silica nanoparticle from rice husk ash for drug delivery application. *Combinatorial Chemistry & High Throughput Screen* **16**(6): 458–462.
29. Bansal V, Ahmad A and Sastry M (2006) Fungus-mediated biotransformation of amorphous silica in rice husk to nanocrystalline silica. *Journal of the American Chemical Society* **128**(43): 14059–14066.
30. Pandey S, Mewada A, Thakur M and Pillai S (2014) Synthesis of mesoporous silica oxide/C-dot complex (*meso*-SiO₂/C-dots)) using pyrolysed rice husk its application in bioimaging. *RSC Advances* **4**(20): 1174–1179.
31. Estevez M, Vargas S, Castaño VM and Rodriguez R (2009) Silica nano-particles produced by worms through a bio-digestion process of rice husk. *Journal of Non-crystalline Solids* **355**(14–15): 844–850.
32. Liou TH, Chang FW and Lo JJ (1997) Pyrolysis kinetics of acid-leached rice husk. *Industrial & Engineering Chemistry Research* **36**(3): 568–573.
33. Mansaray KG and Ghaly AE (1998) Thermal degradation of rice husks in nitrogen atmosphere. *Bioresource Technology* **65**(1–2): 13–20.
34. Ruksudjarit A and Rujijanagul G (2008) Synthesis of SiO₂ nanopowder from rice husk ash. *Advanced Materials Research* **55–57**: 649–652.

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