Characterization of Silica Produced from Rice Husk Ash: Comparison of Purification and Processing Methods

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As a byproduct of the combustion of rice husk to generate energy, rice husk ash (RHA) is formed by silica and carbon, apart from small amounts of other constituents. Several treatments can be used to increase the purity of the silica obtained, or even produce pure silica. The present study tested the efficiency of different techniques to obtain silica, characterizing and comparing the silicas obtained from RHA. A literature review was conducted, and then selected techniques were used to produce silica, which was characterized by XRF, XRD, particle sizing, specific weight, specific surface area, total carbon, and SEM. The literature review showed that most techniques include a pretreatment like acid or alkaline leaching followed by thermal treatment to increase the amount of silica produced by reduction of carbonaceous materials. The results showed that it is possible to produce silica from RHA using simple methods, and that these produced silica with purity above 98%. The treatments that afforded the best results were acid leaching followed by thermal treatment at 800ºC, and alkaline extraction at low temperature, with silica purity of 99.3% and 99.6%, respectively.

Keywords: *rice husk ash, silica, thermal treatment*

1. Introduction

Rice husk and rice husk ash (RHA) are interesting sources of considerable levels of high quality silica, which has several applications[1]. The production of silica at industrial scale is based on mechanical, physical, chemical, and energy-intensive thermal operations at high temperatures using large amounts of acids, generating significant volumes of effluents[2]. In this sense, Ma et al.[3] highlight the fact that the conventional silica production method is based on the reaction between sodium carbonate and quartz at high temperatures. After sodium silicate is formed, silica is precipitated in a reaction with sulfuric acid. But this method also consumes high levels of energy and produces large amounts of liquid effluents and greenhouse gases.

Riveros and Garza[4] discussed the fact that, compared with other silica sources like sand, bentonite, and diatomaceous earth rice husk has very small amounts of contaminants that affect performance in applications requiring high purity, as in the manufacture of solar panels, for instance. Therefore, rice husk is an important source of silica and solar-grade silicon. Jung et al.[5] also claims that rice husk has exclusive nanoporous silica layers, which have developed throughout years of natural evolution of the plant. This is why producing highly reactive silica from rice husk is a simple process with

several advantages, compared with conventional production methods[6].

High purity is required in several applications of silica. Silica is a basic raw material widely used in the semiconductors, ceramics, polymers, and materials industries. This silica is usually produced from quartz fused at high temperatures, which affords to obtain ultrapure polycrystalline silicon and silicon hydride. In addition to environmental and economic advantages, low-energy, simpler methods to obtain pure silica create opportunities for the development of new industrial applications of RHA[7].

Various methods are used to produce larger amounts or pure or ultrapure silica from rice husk or RHA. Table 1 shows a list of studies that looked into different treatments to reduce contamination, improve the properties of RHA, or even produce pure silica.

Table 1 also makes it clear that numerous approaches have been developed to produce high purity amorphous silica with large surface area using rice husk or RHA. Several studies[3,7,10,11] used alkaline extraction sol-gel method, which forms silicates, followed by silica precipitation by reaction with an acid. Kalapathy et al.[7] explained that it is possible to obtain amorphous silica from RHA by alkaline extraction at low temperatures. Also, at pH below 10, the solubility of amorphous silica is very low, but it rises suddenly when pH is increased above that. This solubility profile affords to extract

Table 1. Studies that investigated methods used to obtain and purify silica from rice husk ash.

Study	Objective	Summary of method used	Main results
Sankar et al.[8]	To produce nanosilicon powder from three kinds of rice husk.	• Combustion of rice husk in the open. • Acid leaching • Incineration at 700°C under atmospheric conditions	Spherical, completely amorphous silica particles with large specific surface area and composed only by Si and O from all kinds of rice husk.
Bakar[9]	To investigate the ideal conditions to obtain high purity silica.	• Washing of rice husk with water • Acid leaching • Incineration at 500, 600, 700, 800, and 900 °C for 2 h under atmospheric conditions	All silicas had amorphous particles, and rice husk leached with HCl produced the highest content of silica (99.582%) at 600 °C.
Ma et al.[3]	To develop a new, recycling-based technique to produce silica from RHA.	• Acid leaching of RHA • Extraction in reactor with NH_4F • Acid precipitation of silica	Spherical, completely amorphous silica particles measuring 50 to 60 nm in diameter and containing only Si and O, with yield of 94.6%.
Liou[10]	To investigate the effect of experimental conditions on the characteristics of the nanosilica obtained.	• Leaching of rice husk with water and then HCl • Incineration • Extraction with NaOH, forming silicate • Precipitation	Best results with pH 3, silicate 0.15 M, aging time of 12 h at 50°C, with 99.48% purity silica.
Fernandes[11]	To evaluate the leaching of RHA with NaOH to easily obtain highly reactive colloidal silica.	• Extraction of silica with NaOH 1M • Precipitation using H_2SO_4 drop by drop	The best Si:Na mass ratio was 4:1, and the highly reactive colloidal silica was obtained at low energy investment.
Yalçin and Sevinç[12]	To obtain high purity silica with large specific surface area and to evaluate the competitiveness of silica from rice husk.	• Washing of rice husk with water • Chemical treatments (acid leaching, alkaline leaching) • Incineration at 600°C under different conditions (static oven, argon flow, oxygen flow, air flow)	Amorphous silica particles with maximum specific surface area of 321 m^2/g 99.66% purity, depending on the treatment. The highest purity silica was obtained with prior acid treatment and incineration in oxygen atmosphere.
Kalapathy[7]	Investigate the efficacy of acid leaching of RHA before alkaline extraction and washing of sílica obtained with water.	• Acid leaching • Extraction of silica with NaOH • Precipitation with HCl • Washing with water	Initial acid leaching did not improve purity, the washing with water reduced Na and K levels, and silica yield was excellent when extraction was carried out with NaOH 1 N.
Conradt[13]	To obtain high purity silica with large specific surface area and to evaluate the competitiveness of silica from rice husk.	• Washing rice husk with water • Different chemical treatments (acid leaching, alkaline leaching, and enzymatic digestion) • Incineration at 600°C (static oven, air flow, steam)	Except for leaching with NaOH, all other treatments produced amorphous silica with large specific surface area, and high purity silica was obtained with acid leaching with HCl.
Riveros[4]	To optimize the production of silica from rice husk.	• Washing rice husk with water • Acid leaching • Incineration • Leaching of RHA	Silica with approximate purity of 99.98% with three washing cycles, 6-h acid leaching, 6 leaching cycles with HCl 3% at 90°C, and the main contaminant was Ca (100 ppm).

silica from RHA by solubilization in alkaline medium and subsequent precipitation at lower pH values. Consuming comparatively lower amounts of energy, the alkaline extraction of silica by solubilization is not as costly nor as damaging to the environment as the quartz fusing method.

Other authors used thermal treatments at different temperatures and atmospheres with or without pretreatments[4,8,9,12,13]. Yalçin and Sevinç[12] demonstrated that incineration of rice husk at between 600°C and 800°C preceded by chemical and thermal pretreatment affords to obtain high purity silica.

The authors used different chemical and thermal treatment approaches, but reported that acid leaching pretreatment followed by oxygen-rich incineration and combustion of rice husk produced the highest silica content. The atmosphere with the highest partial pressure of oxygen induced the chemical reduction of carbon, and was more efficient than the other atmosphere settings evaluated.

Despite the numerous approaches to produce and purify silica, RHA is virtually not used for this purpose. Most studies to date have addressed the production of silica directly from

rice husk, not from its ash. Few investigations have looked into the potentialities of RHA as a source of silica, and the samples used generally were RHA produced under controlled laboratory conditions. The production of silica from RHA is very interesting from the industrial perspective, in view of fact that rice husk is normally used as direct source of energy. Yet another important aspect is that the purification method used today normally affects the features of the end product, changing morphology, structures, and reactivity[14].

This study characterized and compared the silicas produced from RHA using different extraction and purification methods. We analyzed the effect of each process on the characteristics of the silicas produced and discuss the advantages of each approach.

2. Materials and Methods

2.1 Materials

The RHA used in this study (called RHA FB) was supplied by a thermoelectric company established in the state of Rio Grande do Sul, Brazil. The company uses rice husk as biomass for the generation of energy by fluidized bed combustion. Briefly, rice husk is loaded into the reactor and instantly burned on a sand bed with ascending air flow at around 700ºC. After combustion, RHA is milled by the producer company.

2.2 Processing and purification of RHA

The different processing and purification techniques were compared for the amount and properties of silica produced based on the literature review of the studies listed in Table 1. The different processing and purification treatments performed are compiled in Table 2.

2.3 Characterization of RHA and silica

Particle sizing was carried out by wet laser diffraction in a particle analyzer (S3500, Microtac) using water as solvent containing one drop of surfactant. Flowrate was 25% and ultrasound time was approximately 30 s. All samples were considered to have a refraction index of 1.6.

Specific weight was determined using a helium pycnometer (AccuPyc II 1340, Micrometrics). After drying in a oven at 105ºC, approximately 5 g of each kind of RHA were placed in a 10-cm³ aluminum sample holder.

Specific surface area of samples was evaluated according to the nitrogen BET (Brunauer-Emmett-Teller) adsorption technique in a surface area analyzer (TriStar II Plus, Micrometrics). Samples were degassed under vacuum at 200ºC for 34 h.

Table 2. Methods used to process and purify silica from RHA FB.

Method and References that treatments performed were based on	Treatment	Abbreviation
Thermal treatment of RHA at different temperatures with no pretreatment[4,8,9,12] and[13]	The samples were heated in a muffle oven in air at 700ºC and 800ºC for 1, 2, and 3 h with heating rate of 5 ºC/min and spontaneous cooling in the oven down to environment temperature.	TT 700 1h TT 700 2h TT 700 3h TT 800 1h TT 800 2h TT 800 3h
Acid leaching followed by thermal treatment[4,8,9,12] and[13]	Washing, contact time, and acid solution were selected based on a literature review. RHA samples were washed with HCl 1M (10 g sample/100 mL acid) for 1 h. RHA was filtered and washed with deionized water upon neutral pH. Samples were dried at 105ºC for 24 h and then treated in a muffle stove in air at 800ºC for 1 h at 5 ºC/min and cooling in the oven down to environment temperature.	LX TT 800 1h
Alkaline extraction at low temperature[3,7,10] and[11]	Extraction based on the solubility of amorphous silica in alkaline pH and precipitation in lower pH. All parameters used were based on a literature review of the articles shown in Table 1. Briefly, 200 mL NaOH 1 M were added to 20 g RHA. The material was boiled for 1 h with constant stirring. After cooling, samples were filtered and the filtrate obtained was titrated with HCl 1 M upon neutral pH. After aging time of 48 h, water was added and the mixture was centrifuged and washed serially. The material was dried in a stove at 105ºC for 48 h.	EXT NaOH

Chemical composition was analyzed comparatively in an energy dispersive X-ray fluorescence (XRF) spectrometer (EDX 720 HS, Shimadzu). The results are expressed as percent by mass calculated over oxide content and normalized to 100% using carbon percent levels. The samples were previously ground and compared with a secondary RHA standard.

Total carbon (TC) was determined using previously dried samples according to the total combustion method at 1,350ºC and infrared detection in a carbon analyzer (SC-144DR, Leco).

The mineralogical composition of samples was analyzed by X-ray diffraction (XRD) in an X-ray diffractometer (X'PERT, Philips) with copper emission lines (CuKα → λ = 1.5418 Å). The experimental conditions were: 40 kV, 40 mA, 2θ scanning range from 5 to 75º, angular step of 0.05º. Peaks were identified using the software X1Pert High Score 2.0a.

The morphological characteristics of gold-plated samples were investigated by scanning electron microscopy (SEM) (EVO MA15, Zeiss) under high vacuum and detection of secondary electrons.

3. Results and Discussion

Table 3 shows particle sizing results (obtained by laser diffraction) and specific weight and specific surface area values. The specific weight of samples, including the control sample without treatment, RHA FB, varied little, between 2.13 g/cm³ and 2.24 g/cm³. Obtaining silica from RHA using different chemical and thermal treatments (500ºC - 800ºC), Yalçin and Sevinç[12] observed that the density of most samples increased with silica contents. In the present study, specific weight of samples did not vary with silica levels. However, this may have been due to the fact that silica levels likewise did not oscillate significantly between RHA samples obtained under the conditions assessed.

Nevertheless, the surface area and mean diameter of silica particles varied significantly using different settings. The positive correlation between surface area with size, shape, and porosity has been discussed in the liteature[15]. The results we obtained here show that specific surface area was influenced less by particle size than by porosity and shape, since the sample with the longest mean diameter was also the one with the largest surface area. At 290 m²/g, the sample obtained by sol-gel method at low temperature (EXT NaOH) had a significantly large surface area, compared to other samples. This may be associated with the sol-gel process used to produce EXT NaOH. Large surface area is desirable in many applications, such as in the reinforcement of polymer compounds. At any rate, it is necessary to consider the chances that, at approximately 205 µm, the diameter of the particles obtained by the EXT NaOH method reached that length because the material is an agglomerate, which may have influenced surface area due to the markedly irregular shape of particles.

The results of the present study also indicate that specific surface area decreases with the temperature of thermal treatment. Surface area is reduced with thermal treatments. The untreated sample had specific surface area of 11.35 m²/g. Yet, independently of time, specific surface area of silica particles obtained at 700ºC was around 2.8 m²/g, while for those obtained at 800ºC the values ranged between 1.4 m²/g and 1.7 m²/g. In previous studies by our research group[16], thermal treatments at even higher temperatures (1000ºC, 1200ºC, and 1400ºC) reduced specific surface area of RHA even more, reaching 0.44 m²/g. Such finding was also observed by Bie et al.[17] and Kapur[18], who showed that the surface area of RHA is influenced by (i) combustion temperature (the higher the temperature, the smaller the resulting specific surface area); (ii) the amount of potassium present (high potassium levels reduce specific surface area and increase mean diameter, since the element works as fusing agent); and (iii) the material structure (crystalline silica has smaller surface area, compared to amorphous silica). In the present study, the sample previously leached with acid solution (LX TT 800 1h) had slightly larger specific surface area, compared with the other samples treated at the same temperature. So, it may be said that prior acid leaching increased porosity, or that the larger surface area values recorded are associated with potassium levels.

Table 3. Specific weight, specific surface area, and grain sizing of thermally treated RHA samples.

Sample	Mean diameter (µm)	Specific weight (g/cm³)	Surface area (m²/g)
RHA FB	19.56 ± 0.49	2.22 ± 0.0028	11.35 ± 0.21
TT 700 1h	18.53 ± 0.04	2.23 ± 0.0030	2.83 ± 0.046
TT 700 2h	18.01 ± 0.10	2.23 ± 0.0017	2.78 ± 0.047
TT 700 3h	17.72 ± 0.01	2.24 ± 0.0017	2.74 ± 0.045
TT 800 1h	22.55 ± 0.23	2.23 ± 0.0011	1.70 ± 0.042
TT 800 2h	22.60 ± 0.30	2.21 ± 0.0026	1.70 ± 0.040
TT 800 3h	22.78 ± 0.11	2.22 ± 0.0036	1.46 ± 0.039
LX TT 800 1h	20.10 ± 0.59	2.20 ± 0.0034	2.97 ± 0.017
EXT NaOH	205.10 ± 4.60	2.13 ± 0.0162	290.03 ± 1.21

We also observed that the mean diameter of particles increased with temperature. The treatments at 700ºC and 800ºC produced particles with mean diameters of approximately18 µm and 22.6 µm, respectively. This longer mean diameter may result from the mild pre-fusion of particle surfaces, possibly due to the presence of potassium. Mean diameter of particles of the sample pretreated with acid leaching was slightly shorter than that of the other samples treated at 800ºC, which may also be associated with the lower potassium content in this sample.

The sample EXT NaOH had mean diameter of 205.10 µm. In addition, the specific surface area of EXT NaOH particles was of another order of magnitude, not affording comparison with the other treatments performed in this study.

The results of the chemical characterization by XRF and TC levels are shown in Table 4.

All treatments produced RHA with high silica content and low TC levels. The thermal treatments used did not affect the chemical composition of samples consistently. Potassium was the main contaminant, varying between 0.48 and 0.70. Also, high treatment temperatures produced samples with lower TC. But the sample obtained by alkaline extraction at low temperature (EXT NaOH) had high silica level, with Cl as a contaminant. This is explained in view of the treatment adopted, which included silica precipitation with HCl. Additional washing with deionized water may help remove this residual contaminant. It is also possible to see that, at both 700ºC and 800ºC, incineration time did not substantially influence composition, meaning that the period of 1 h of thermal treatment is enough to produce desirable results.

The highest silica content was obtained for the RHA submitted to the EXT NaOH treatment (99.61%), followed by the one submitted to the LX TT 800 1h protocol (99.25%). The other thermal treatments at 700ºC and 800ºC produced similar contents of silica (between 98.7% and 98.9%). The two samples that had the highest silica levels were also those with the lowest TC content, EXT NaOH (0.10%) and LX TT 800 1h (0.09%).

Percent silica levels obtained in this study are considerably high, compared with data published in the literature (Table 1). The main reason may be the fact that the studies that obtained the highest silica contents, namely 99.98%[4], 99.66%[12], 99.58%[9], and 99.48%[10] used rice husk as raw material, not RHA. In fact, considering the studies listed in Table 1, most[4,8,9,10,12,13] obtained silica from rice husk, while a smaller number[3,7,11] used RHA.

Ugheoke and Mamat[19] explain that one of the reasons why it is more difficult to obtain silica with purity above 97% from rice husk by direct incineration is the presence of metallic contaminants, especially potassium and sodium oxides. These compounds affect the surface of silica particles, increasing their surface area and reactivity, and increase silica crystallization rate. Acid leaching followed by thermal treatment (LX TT 800 1h) induced the production of silica with the lowest carbon level (0.09%). Krishnarao et al.[20] explained that acid leaching prevents the formation of black particles, since it removes potassium, the main agent responsible for the presence of unburnt carbon in ash. The authors showed that potassium works as catalyst in the crystallization of silica and, when temperature rises above the dissociation temperature of K_2O (approximately 347ºC), the surface of ash particles begins to melt, blocking the transportation of oxygen and CO_2 and increasing the amount of unburnt carbon. Therefore, the low potassium level (0.48%) found in this sample may explain the smaller amount of carbon.

In this sense, a pretreatment strategy to remove metallic contaminants affords to obtain high purity silica with particles of larger specific surface area, compared with direct combustion[19].

Table 4. Chemical analysis by energy dispersive X-ray fluorescence spectrometry and total carbon of samples (TC: total carbon; ND: not detected).

Sample/ Composition	RHA FB	TT 700 1h	TT 700 2h	TT 700 3h	TT 800 1h	TT 800 2h	TT 800 3h	LX TT 800 1h	EXT NaOH
SiO_2	96.73	98.71	98.79	98.68	98.87	98.94	98.99	99.25	99.61
Al_2O_3	0.09	0.09	0.07	0.07	0.04	ND	0.02	0.09	ND
P_2O_5	0.23	0.07	ND	0.06	0.04	ND	ND	ND	ND
SO_3	0.06	0.06	0.06	0.05	0.07	0.06	0.05	0.04	0.06
Cl	0.01	ND	ND	ND	ND	ND	ND	ND	0.08
K_2O	0.69	0.72	0.72	0.73	0.71	0.71	0.71	0.48	0.06
CaO	ND	ND	ND	ND	ND	ND	ND	ND	ND
MgO	ND	ND	ND	ND	ND	ND	ND	ND	ND
TiO_2	ND	ND	ND	ND	ND	ND	ND	ND	ND
MnO	0.01	0.02	0.02	0.02	0.02	0.02	0.02	0.04	0.10
Fe_2O_3	ND	ND	ND	ND	ND	ND	ND	ND	ND
ZnO	ND	0.01	0.01	0.01	0.01	0.01	0.01	ND	ND
TC	2.18	0.32	0.33	0.38	0.25	0.26	0.21	0.09	0.10

Figure 1 presents the diffractograms of the samples analyzed. Several studies have used different time and temperature conditions to produce amorphous or crystalline silica. However, most authors agree that amorphous silica is produced at temperatures below 800ºC. Several studies have shown that higher temperatures induce the formation of crystalline dots.[18,19,21,22]
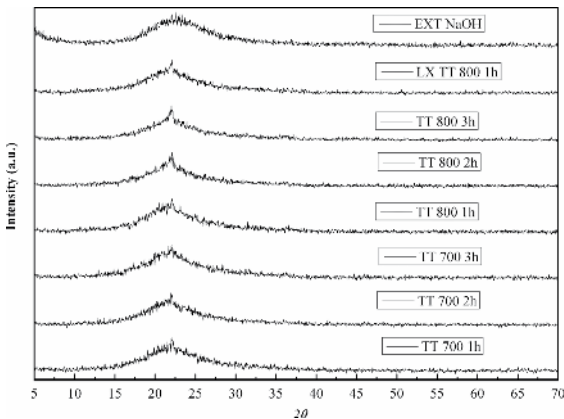

Figure 1. Diffractogram of the treatments used.

The results of the present study confirm previous findings, since all treatments led to the generation of predominantly amorphous silica. Kapur[18] demonstrated that the cristobalite phase starts at 800ºC. The results of the present study confirm the findings of the author, since, although they are predominantly amorphous, the samples treated at 800ºC presented the cristobalite phase as main peak. The silica obtained by alkaline extraction at low temperature (EXT NaOH) was completely amorphous. This is due to the fact that NaOH solubilizes only amorphous silica, which is subsequently precipitated. The sample pretreated with acid leaching (LX TT 800 1h) did not differ from the sample that was incinerated at the same temperature but without previous leaching (TT 800 1h).

In a study that tested various temperatures between 500ºC and 900ºC to incinerate rice husk, Bakar et al.[9] used raw samples and previously leached ones, and obtained amorphous silica. However, the authors highlighted that the sharpness of the peak increases with combustion temperature, mainly for non-leached samples, indicating that silica crystallization starts at around 900ºC and that the removal of alkaline species during acid leaching is an obstacle to the eutectic reaction of silica. The behavior predicted in Figure 1 is similar to the findings reported by Bakar et al.[9], since the sample heated to 800ºC for 3 h had a sharper peak, compared to the others.

Figure 2 shows the SEM microphotographs of silica obtained from RHA samples subjected to the different treatments tested. The silica particles produced from all samples were irregular and jagged. Can be observed agglomerates of particles below 10 μm. Although the different pretreatments used affected the chemical composition of the silicas obtained,
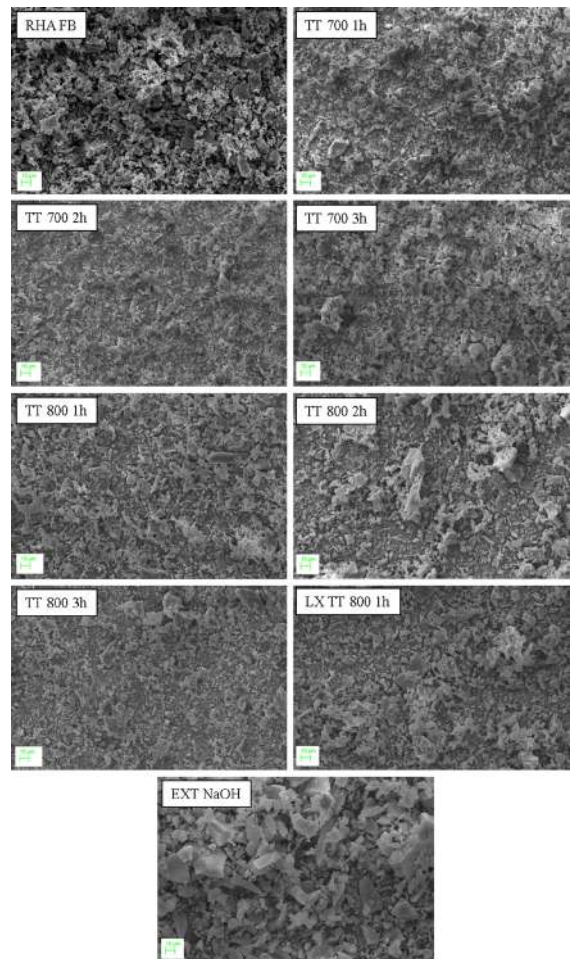

Figure 2. Microphotographs obtained by SEM of the different samples (Original magnification 1,000x).

it seems that the structure and the morphology of these particles was not altered.

4. Conclusion

The results of the present study show that it is possible to produce silica from RHA using simple processes. The various production methods tested afforded to obtain silica with purity above 98%, especially acid leaching followed by thermal treatment and sol-gel alkaline extraction at low temperature, when silica contents were between 99.3% and 99.6%, respectively. Alkaline extraction at low temperature also produced silica particles with large specific surface area (approximately 290 m²/g), compared with the other treatments evaluated, and proving to be the most advantageous technique.

The purity values observed, between 98.7% and 99.6%, are high, compared with data reported in the literature, especially considering the fact that most studies extracted silica directly from unburnt rice husk.

5. Acknowledgements

6. References

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