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Extraction of Silica from the residue of Thermoelectric Power Plants

Brazil presents the second largest coal reserves in Latin America, whose exploration is mainly focused on electricity generation. The present study was carried out to systematically evaluate the influence of various physical and chemical factors on the extraction of pure amorphous silica precipitated from coal-fired bottom ash. The coal bottom ashes (CBA) is a disposable waste from thermoelectric plants, which basically consists of oxides of silicon (80-60%), aluminium (25-20%) and iron (10-2%), being considered as raw material for silica production, by the sol-gel process. The CBA characterization was carried out by determining moisture, elemental composition (XRF), amorphism (XRD) and granulometry. The process evaluation tookplace in two stages: the quantitative evaluation that wasrelated to the quantification of the amount of silica obtained and the process variables (NaOH concentration and temperature); and the qualitative evaluation the silica extracted. The extracted silica particles were characterized by TG, BET, XRD.Silica and aluminium contents were obtained by gravimetric analysis. Among the factors analysed, the concentration of sodium hydroxide and temperature interference were the most important. Likewise, the repeat process for the same ash sample (sequential extraction) proved to be more effective than increasing the NaOH concentration. The obtained yield was 51.34%. The silica is 89.4% pure, when 5,63% of impurities are related to aluminium coxide.

Keywords: Coal-Fired Bottom Ash; Amorphous Silica; Xerogel; Aquagel.

Extração de sílica a partir do resíduo de usinas termelétricas

O Brasil apresenta a segunda maior reserva de carvão da América Latina, cuja exploração se concentra principalmente na geração de eletricidade. O presente estudo foi realizado para avaliar sistematicamente a influência de vários fatores físicos e químicos na extração de sílica pura amorfa precipitada a partir de cinzas de carvão. As cinzas de fundo de carvão (CBA) são resíduos descartáveis de usinas termelétricas, que consistem basicamente em óxidos de sílicio (80-60%), alumínio (25-20%) e ferro (10-2%), sendo considerados matéria-prima para produção de sílica, pelo processo sol-gel. A caracterização da CBA foi realizada por determinação da umidade, composição elementar (XRF), amorfismo (XRD) e granulometria. A avaliação do processo correu em duas etapas: a avaliação quantitativa, relacionada à quantificação da quantidade de sílica obtida e das variáveis do processo (Concentração e temperatura de NaOH); e a avaliação qualitativa da sílica extraída. As partículas de sílica extraídas foram caracterizadas por TG, BET, DRX. Os teores de sílica e alumínio foram obtidos por análise gravimétrica. Entre os fatores analisados, a concentração de hidróxido de sódio e a interferência de temperatura foram os mais importantes. Da mesma forma, o processo de repetição para a mesma amostra de cinza (extração sequencial) mostrou-se mais eficaz do que aumentar a concentração de NaOH. O rendimento obtido foi de 51,34%. A sílica é 89,4% pura, quando 5,63% das impurezas estão relacionadas ao óxido de alumínio.

Palavras-chave: Cinzas de Carvão; Sílica Amorfa; Xerogel; Aquagel.

Topic: Engenharia de Materiais

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INTRODUCTION

Mineral coal constitutes the world's largest source of energy. Approximately 40% of the planet's electrical energy is generated by thermoelectric coal-fired power plants (MCHUGH, 2016). The environmental impact arising from the generation of electricity in thermoelectric coal is enormous, which has increased interest in research aimed to minimize the volume of waste and the impacts related to its disposal.

According to data from the American Coal Ash Association (ACAA, 2017), the United States, the world's largest consumer of coal products, generated more than thirty-five million tons of ash in 2016, of which 3.425.057.691 tons was bottom ash. However, despite the expressive numbers, these figures correspond to less than 40% of the volume of coal bottom ash produced in the country.

Brazil presents the second largest coal reserves in Latin America, whose exploration is mainly focused on electricity generation. Brazilian coal has higher content of mineral matter, which is responsible for the generation of a greater amount of inorganic solid wastes (ashes). It is estimated that the volume of ash generated by Brazilians thermoelectric plants is equivalent to the volume of charcoal burned (ROHDE et al., 2006).

In the thermoelectric plants, two types of ashes are generated: fly ash and bottom ash. Fly ash consists of an extremely fine spherical particles that are carried along the chimney, by the flow of gases of the combustion and collected by some filter systems. Bottom ash is deposited on the walls and at the bottom of the coal furnace, consisting of larger, unformed particles with dimensions similar of sand grains which are removed by pressurized water flow (ROHDE et al., 2006).

Despite the analogous elementary constitution, the ashes present a distinct reactivity: fly ash is classified as "artificial pozzolan" (ABNT, 2014), being used in Brazil in the production of Portland cements and other related materials. The bottom ash presents a lower pozzolanic reaction in which almost all the production is being discarded, either in open pit deposits, in sedimentation ponds or in mine pits (REPETTE et al., 2011; RODHE et al., 2016).

Thus, this study investigates the viability of coal-fired ash (CBA), a disposable waste which basically consists of oxides of silicon (80-60%), aluminium (25-20%) and iron (10-2%) (ROHDE et al., 2006) being considered as raw material for silica production. Silica, in addition of being the precursor material for a variety of inorganic and organometallic materials, has a wide application in industry, from detergents and glues to pharmaceuticals, chromatography column filling, dental materials, electronics and ceramics (SRIVASTAVA et al., 2013).

The conventional process for obtaining silica from quartz crystals (sand) involves multiple steps and very high temperature and pressure. These procedures imply to great environmental impact and are quite expensive. On the other hand, the sol-gel method has been widely applied in the production of silica, glass and ceramic materials, due to its ability to form pure and homogeneous products in moderate conditions (RAHMAN et al., 2012).

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The excellent results presented in the extraction of silica from rice husk ash, awaste from rice production (CHAKRAVERTY et al., 1991; KALAPATHY et al., 2000; 2002; YALÇIN et al., 2001; MA et al., 2012; ZULKIFLI et al., 2013; HAQ et al., 2014; MORSY et al., 2014; GU et al., 2015B; SOLTANI et al., 2015; LIU et al., 2016) also other agricultural products as sugarcane bagasse (AFFANDI et al., 2009; FAIRBAIRN et al., 2012), corn cob and bark (MOHANRAJ et al., 2012; OKORONKWO et al., 2013; SHIM et al., 2015) and wheat bark (JAVED et al., 2011) have motivated researches on the viability of obtaining SiO2 particles through the method (sol-gel), with a relative degree of purity and very low consumption of energy. However, the adoption of the sol-gel process for the extraction of silica from bottom ash has not been investigated yet.

The present study was carried out to systematically evaluate the influence of various physical and chemical factors on the extraction of pure amorphous silica precipitated from CBA. The results allow a rational approach to the development and production of highly purified silica to meet the individual requirements by the sol-gel process.

MATERIALS AND METHODS

This study employs an analysis on the manufacturing, main process variables and characterization of silica obtained from CBA by the sol-gel method. This process is based on the alkaline solubilization of the amorphous silica constituent of the ash followed by precipitation and dissolution on a laboratory scale. The methodology adopted is presented below, as well as the description of the reagents and analyses used in the whole process to obtain the silica and evaluation of the results.

The sol-gel method adopted in this research uses sodium silicate (Na2SiO3) obtained from coal ash by hydrotreating with NaOH as metallic alkoxide and hydrochloric acid (HCl) as a mineral acid. All reagents used were of analytical grade. All procedures were performed with distilled water in triplicate. The CBA used in this research comes from Unit III of the Presidente Médici Power Plant (Candiota – RS, Brazil). The experimental work, shown in Figure 1, can be divided into three stages: a) Characterization and preparation of CBA; b) Sol-gel process; and c) Evaluation of the process.

The first step of the experimental program focused on the characterization of the material and pretreatment of the sample. The CBA characterization consisted of determination of moisture content, grain size, elemental composition (XRF) and degree of atomic organization (XRD). The pre-treatment consisted of drying and milling the sample.

The sol-gel process consists of the solubilization of the silica contained in the CBA through contact with NaOH solution (hydrolysis), forming a solution of sodium silicate, which with subsequent titration of HCl, becomes a biphasic mixture of silica gel and sodium chloride solution. Depending on the density difference, the substances are easily separated. Silica powder (xerogel) is obtained by dehydration of the gel. Residual sodium in both the gel and xerogel is eliminated by washing. The evaluation takes place in two stages: the quantitative evaluation that is related to the quantity of silica obtained and the process variables (NaOH concentration and temperature) and the qualitative evaluation, which consists of the characterization of the silica extracted.

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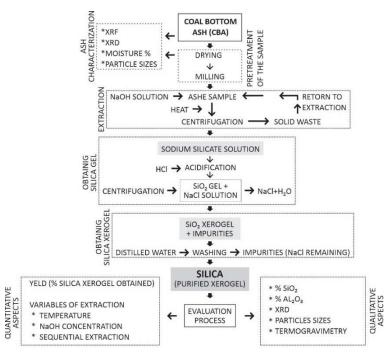


Figure 1: Flowchart of the silica extraction process from CBA.

Characterization of the CBA and pre-treatment of the sample

The pre-treatment process of the CBAconsisted of drying and milling the sample. The characterization was carried out by determining the degree of moisture, elemental composition (XRF), degree of amorphous (XRD) and granulometry. The moisture content of the ash sample was determined by the weight difference between the wet sample and the dried sample in an oven at 80 °C for 24 hours - according to the Brazilian Standard for determination of moisture in coal–ABNT NBR16508 (ABNT, 2016).

The granulometric composition was defined through a set of sieves, with mechanical agitation – ABNT MN 248 (ABNT, 2003). X-ray Fluorescence (XRF) analysis was performed withRigaku's RIX 2000 X-Ray fluorescence spectrometer, thesamples were milled and sieved #200, then pressed in the sample holder.Finally, the X-ray diffraction (XRD) of CBA was performed in a MD-10 X-ray minidiffractometer, CuKa radiation (RADICON, Russia), using an acceleration voltage of 25 kV and current of 400 uA. The diffraction angle was scanned from 10° to 75° 20, at a rate of 3.25s.min⁻¹.

The CBA preparation consisted of homogenization, quarrying, drying (85 °C for 24 hours) and grinding of the sample. Approximately 10 kg of CBA were milled in a ceramic ball mill for 8 hours, with a ratio of 5: 1 grinding / ash mass, 40 rpm rotational speed and grinding bodies with a diameter varying between 10 and 20 mm based onPouey (2006). The final particle size was determined by laser diffraction in a CILAS equipment, Particles Size Analyzer, model CILAS 1180Liquid(CILAS, Orleans France). The analysis range was from 0.04 to 2500 μ m and the grain size of the milled ash reachedD_{mean}= 11.09 μ m, D₅₀= 6.36and D₉₀= 28.05 μ m.

Obtaining sílica

Theprocess shown in Figure 1 was adapted from the method adopted by Srivastava et al. (2013) and can be subdivided into 3 stages: extraction, gel acquisition and xerogel production. In the first stage, the mixture (CBA+ NaOH solution) was heated in a water bath in a 500 ml Erlenmeyer flask for 1 h, capped [10] and under constant stirring for complete dissolution of silica and obtaining the sodium silicate solution. After this period, the material was cooled to the room temperature, centrifuged and filtered in 6 batteries for 5 minutes at 3200 rpm and then intercalated with distilled water wash. The solid residue of the filtration returned to the process until exhaustion (sequential extractions).

In the second stage, the solution containing the silica was transferred to another beaker for the formation of the gel. The formation of the gel occurs when the pH of the solution is reduced to valuesbelow 9 by the addition of HCl under constant agitation (KALAPATHY et al., 2002). At pH 7, the solution was allowed to stand to form the gel. After the resting period ('aging') of about 24 hours the soft gel was gently broken and the sludge was centrifuged at 3200 rpm for 5 minutes, the solutionwas discarded and the gels was transferred to a beaker with distilled water.

In the third and final step, the wet centrifuged gel wasplaced in an oven at 50 °C for 72 hours to obtain the dried silica (powder) known as "xerogel" (BRINKER et al., 1990). The silica xerogel was further flushed with distilled water for effectively removing mineral impurities from the silica (OKORONKWO et al., 2013; PRASAD et al., 2012).

The efficiency of the washing process and the consequent absence of residual sodium chloride was signalled by the addition of a few drops of silver nitrate solution to the water discarded from the silica wash. Silver nitrate immediately reacts with chloride ions, precipitates and makes the mixture cloudy. The washing process wasrepeated until the discarded water didnot contain chloride ions. After washing the silica is placed again in the oven to dry, until the constancy of the mass.

Process evaluation

The results wereanalysed under both quantitative and qualitative aspects. The quantitative evaluation wasbased on the relation between the amount of residue (CBA) used and the percentage of silica extracted, that is, the yield obtained by Equation (1).

Yield (%) =
$$\frac{m1 \times Si1\%}{m2 \times Si2\%}$$
 (1)Wherem1 = mass of silica extracted

m1 = mass of silica extracted m2 = CBAmass Si1% = percentage of silica contained in the silica extracted Si2% = percentage of silica contained in the ash (XRF)

Some studies (AYEGBA et al., 2015; KALAPATHY et al., 2000; LIU et al., 2016; MOHANRAJ et al., 2012; SRIVASTAVA et al., 2013) point out that the main factors influencing the yield during the extraction process are the temperature and NaOH concentration. Due lack of similarities in the parametersadopted in

previousresidue extraction surveys, it was decided to study the influence of these parameters on the yield of the CBA extraction process. Thus, in the extraction stage, the following variables were adopted: a) **NaOH concentration**. Two NaOH/CBAmolar ratio concentrations were tested. The first concentration (NaOH/CBAratio = 0.4), common in researches with plant ashes residues (KALAPATHY et al., 2000; SHIM et al., 2015) and the second (NaOH/ash ratio = 1.2), used in corn cob ash (MOHANRAJ et al., 2012); b) **Sequential extraction** ($3x0.4 \ge 1x1.2$) is demonstrated the efficiency of the repetition in leaching analyses and extractions studies. Analysis of the efficiency of sequential extractions seeks to explore ways of reducing the volume of reagents and improving the yield of the process; c) **Temperature** (85 °C and 95 °C). Srivastava et al. (2015) demonstrated the direct influence of extraction temperature on yield in his studies with perlite. Initial tests indicated an even greater interference in silica extraction from CBA. This analysis seeks ways of evaluating cost/benefit by increasing the extraction temperature by 10 °C.

The qualitative evaluation of the silica was determined by the characterization of the silica obtained. The moisture content was determined by thermogravimetry (TG); the particle size was assessed by specific surface analysis (BET); the silica and aluminium contents were obtained by gravimetric analysis and the degree of amorphous of the silica produced was evaluated by XRD.

Regarding the instruments used in the analyses, for TG analysis, an analytical thermobalance model STA 409 PG (NETZSCK, Selb, Germany) under flow rate of 60ml.min⁻¹ of nitrogen and heating rate of 10°C.min⁻¹ up to 1000 °C was used. The specific surface of the sample was evaluated by Brunauer, Emmett and Taller (BET) isotherms in ASAP 2420 apparatus under the following conditions: a) Degassing temperature: 60 °C; b) Heating rate: 10 °C / min; c) Data collection for BET equation: 6 equidistant points in the range of 0.05 to 0.3 (P / Po) and d) Pressure setpoint (Degassing): 1 Pa. Laboratory environmental conditions during BET analysis were temperature of 23 °C² 5 °C and relative humidity: 55% ² 25%. The X-ay diffraction (XRD) analysis of silica was performed on the same instrument and followed the procedures adopted for ash characterization and finally, the determination of aluminium and silicon contents were determined by gravimetry (ABNT, 2012).

RESULTS AND DISCUSSION

Ash Characterization

The results indicate 33% ofmoisture content in the CBA. The high moisture content is attributed to the process of residue extraction from the interior of the furnace. Naturally, the percentage of moisture varies according to the place of sample collection. The granulometric analysis of CBA confirmed the results of previous researches (MALLMANN, 1996; PIRES et al., 2004; ROHDE et al., 2006; BRAGANÇA et al., 2008); in which the CBApresents the granulometric curve in the thick zone, besides presenting approximately 20% largergrains than 4.8mm, according to the graph shown in Figure 2.

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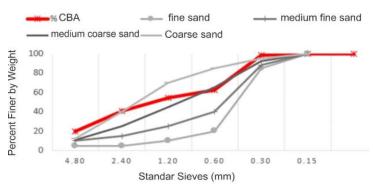


Figure 2: Particle size curve of the CBA. Fonte: ABNT (2005).

The elemental composition of the CBA determined byXRF analysis are presented in Table 1. The table also presents the average XRF results of the CGTEE (Presidente Médici coal fired thermoelectric power plant) ashes from previous researches (MALLMANN, 1996; ROHDE et al., 2006; BRAGANÇA et al., 2008).

	SiO2	Al ₂ O ₃	Fe ₂ O ₃	CaO	K₂O	MgO
СВА	54,96	34,08	5,45	2,15	1,66	0,30
Previous results	65, 47 ^{+4,96} -6,57	21,81 ^{+14,20} 5,18	7, 24 ^{+8,46} -4,88	0,96 ^{+0,21} -0,36	$1, 11^{+0,76}_{-1,11}$	0, 50 ^{+0,25} _{-0.30}
	TiO ₂	CI	SO₃	MnO	Cr ₂ O ₃	CuO
СВА	0,62	0,57	0,07	0,05	0,03	0,01
Previous results	0, 45 ^{+0,35} -0.45	-	0 , 16 ^{+0,27} _{-0.16}	0 , 01 ^{+0,04} _{-0.01}	-	-

Table 1: Results of XRF analysis of CBA, % dry basis.

*Elemental classification of the bottom ashes of PresidenteMédici thermoelectric plant published in other studies (MALLMANN, 1996; ROHDE et al., 2006; BRAGANÇA et al., 2008).

The CBAhad higher aluminium content (34.08%) than expected and slightly lower silica content (54.96%) than that mentioned by previous researchers. The iron content result corresponds to the expected range (between 2.35 and 15.70). The differences between the results are attributed to the mineralogical characteristics of mineral coal whose composition varies. The XRD diffractogramshown in Figure 3 reveals a high degree of amorphicity, characterized by the hump in 2θ between the angles of 15° and 45°. Acute and sharp peaks indicate the existence of chrystals essencially consisting of Quartzand Hematite.

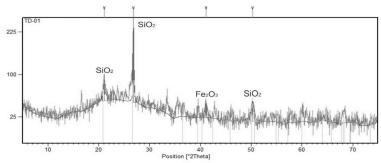


Figure 3: XRD analysis results of the bottom ash coal (CBA).

The XRD results are also consistent with the results of previouspublications, which demonstrate high degree of amorphicity and the presence of Quartz crystals and mullite (ROHDE et al., 2006). The degree of amorphismof the ashes is directly related to the characteristics of the burning and cooling process of the ashes. In the case of charcoal ashes from thermoelectric plants with a well-monitored burning process, amorphous is a constant characteristic.

Evaluation of Process Variables

Concentration NaOH

The results indicate that the production of the extracted materialincreases as the NaOH concentration increases. Figure 4 illustrates the influence of the concentration of sodium hydroxide solution on the percentage of silica extracted. Two concentrations were chosen for the sodium hydroxide solution. The first column of the graph shows the yield corresponding to the solution of 1mol.L⁻¹ (NaOH/ash molar ratio = 0.4), adopted in previous research (RAMESH, 2016; SHIM et al., 2015).The second column corresponds to the yield obtained with the more concentrated solution corresponding to 3mol.L⁻¹ (NaOH/ash ratio= 1.2) adopted by Mohanraj et al. (2012). The results demonstrate the direct relationship between concentration and yield.

Sequential Extractions

The comparison between the cumulative yield of three consecutive extractions (NaOH/ash ratio = 0.4) and the yield of a single extraction, of three higher concentration (NaOH/ashratio = 1.2) indicates that sequential extractions have higher efficiency. Thus, the graph shown in Figure 5, records a single extraction with 1mol.L⁻¹ NaOH solution (NaOH/ash ratio = 0.4), along with the yield of 10.9%, and with triple concentration, a yield of 23.20% was obtained. However, when repeated the process 3 times (sequential extractions), instead of tripling the concentration with 1mol.L⁻¹ solution, the total yield was higher (24.93%).

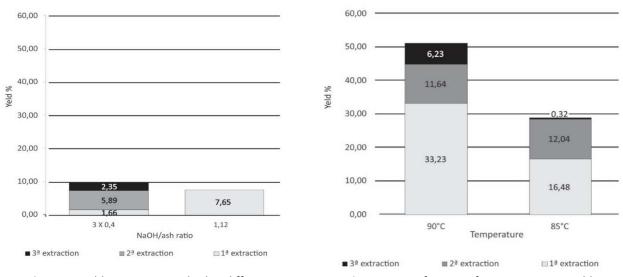
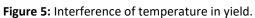


Figure 4: Yield Extraction method at different concentrations of NaOH.

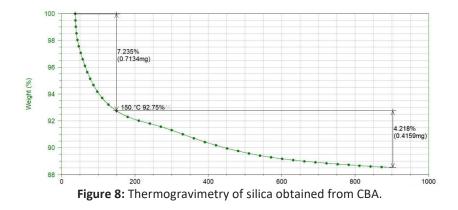


Temperature (85 °C and 95 °C)

Figure 6 correlates the results of multiple extraction with concentrated solution (3mol.L-1). The results obtained point out the direct interference of the temperature in the efficiency of the process: anincrease in temperature by 5 °C reflects in an increase in the production of 12.8%.

Characterization of silica obtained

Silica moisture(interstitial water) was determined by the thermogravimetry test, whose result is shown in Figure 8.



The mass loss up to approximately 150 °C is attributed to the moisture of the air, absorbed by the silica in the sample handling. The mass loss of approximately 4,2% can be attributed to the hydroxyls of xerogel silica (inside the structure). The size of the silica particles was determined through the specific surface. The silica obtained from the CBA had a specific surface of $153m^2g^{-1}$. This result surpassed the initial expectation of having a higher value than those reported in studies with silica from agricultural residues (AFFANDI et al., 2009; SHIM et al., 2015; SUN et al., 2001) whose BET method results range from 20 to $152m^2g^{-1}$.

With respect to the chemical composition of the silica obtained, the percentages of silica and aluminium oxide determined by gravimetry correspond respectively to 89.4% purity (SiO₂) and 5.63% Aluminium oxide (AI_2O_3). The amorphicity of the silica was evaluated by XRD. The X-ray diffraction of the extracted silica from CBA is shown in Figure 9, where a wide hump, centred at 20, between 20° and 30°, is associated with amorphous silica. The absence of sharp inflection indicates the absence of crystalline phases.

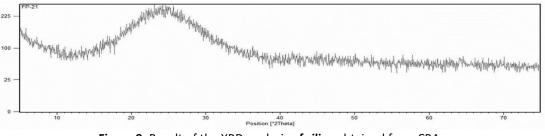


Figure 9: Result of the XRD analysis of silica obtained from CBA.

CONCLUSIONS

This study reveals the feasibility of obtaining high purity silica from CBA with minimal energy outflow using the sol-gel method. The results show a great opportunity to use the ashes to constitute raw material to obtain silica gel with a low aluminium content. Among the factors analysed, the concentration of sodium hydroxide (NaOH/ash ratio) and temperature interference were the most important. Likewise, sequential extraction proved to be more effective than increasing the NaOH concentration. The yield obtained for triplicate extractions of 3mol.L⁻¹ (NaOH/ash ratio = 1.2) was 51.34%. The silica obtained in this study presents 89.4% purity, 5,63 % Al₂O₃ and 4,97% H₂O and others impurities. This researchwas the first step in the development of a more comprehensive study on the idealconditions for silica extraction from coal-fired bottom ashfrom thermoelectric power plants. Alsoshowed the pH interferences on the silica characteristics produced.

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