CIWC-2 2020

sciforum

Conference Proceedings Paper

Synthesis of Silica Particles from Sugarcane Bagasse Ash for Its Application in Hydrophobic Coatings

Jesús A. Pérez-Casas [1,*], Antonio A. Zaldivar-Cadena [2], Anabel Álvarez-Mendez [1], Juan J. Ruiz-Valdés [1], Salomé De la Parra [1] and Astrid I. Sánchez-Vázquez [1]

[1] Universidad Autónoma de Nuevo León, Facultad de Ciencias Químicas, Avenida Pedro de Alba s/n, San Nicolás de los Garza, 66455, N.L., Mexico

[2] Universidad Autónoma de Nuevo León, Facultad de Ingeniería Civil, Avenida Pedro de Alba s/n, San Nicolás de los Garza, 6455, N.L., Mexico

* Correspondence: jesus.apc91@gmail.com

Received: 19 February 2020; Accepted: 12 May 2020; Published: 13 May 2020

Abstract: Wastes like sugarcane bagasse ash (SCBA) can be used as raw material in the ceramics, by the elaboration of bricks and tiles, and glass industry due its high amount of silica in its composition (>70 %). Another application for the SCBA is the synthesis of metallic silicates. In this work is studied the synthesis of sodium silicate with SCBA as main raw material and the future application of the sodium silicate for the preparation of silica particles to create hydrophobic surfaces for ceramic materials to prevent its erosion. The sodium silicate synthesis was carried out by the thermochemical method with batches of ash and sodium carbonate in a 1:1 sodium oxide-silicon oxide molar ratio. The thermal treatment was in an electric furnace at 800 °C for 8 hours, then for the synthesis of the silica particles, the sodium silicate was dissolved in water and there was added methanol in a 3:2 water methanol volume ratio, the solution was aging for an hour to create the bond Si-OH, finally TEOS was added and the solution was stirred for 2 hours to originate a hydrophobic and hydrolytically resistant siloxane by the displacement of the H in the Si-OH bond. The application of the solution was by the spray-coating method over substrates of concrete and red clay with an application of 10, 15 and 20 layers. The hydrophobicity was evaluated with the water contact angle test, with results of contact angles above the 110°, thus demonstrating the capacity of a waste for the generation of coatings to prolong the useful life of building materials.

Keywords: Sugarcane bagasse ash; hydrophobic; sodium silicate

---

Introduction

One of the most common problems in buildings and monuments surface is the erosion due the rain, this slowly dissolves the materials or by the propagation of water across the cracks allowing the corrosion of the internal structure [1,2]. There are alternatives such as covering the materials with hydrophobic materials to prevent the impact over the external structure. Treated silica particles are one of the main selected material for this application due its capacity to create siloxanes over the ceramic substrates [3]; siloxanes are known for its hydrolytically stability and it´s hydrophobic behavior making them a good alternative to protect buildings and outdoor sculptures [4]. In this work to generate silica particles was used a solution of sodium silicate synthetized from sugarcane bagasse ash (SCBA), the main waste of the sugar industry, as main raw material. The SCBA has been studied as additive for the elaboration of building materials nevertheless, only 10 to 15% are allowed before the collapse of the materials due the high porosity [5].

Materials and methods

For the sodium silicate synthesis, it was used sugarcane bagasse ash form the Xicotencatl sugarmill located at the south of the state of Tamaulipas, Mexico. The SCBA was analyzed by an elemental analyzer (Perkin Elmer Inst. Series II 2400 CHNS-O) to quantify the volatile matter and then was calcinated at 900 °C for two hours [6]. The resulting ash was analyzed again by elemental analysis to make sure the elimination of carbon and X-Ray fluorescence (XRF) (Panalytical Epsilon 3) to quantify the inorganic matter. The ash was then milled and sieved till standard mesh #100 (150 μm) and finally leached with citric acid at 2% for 2 hours and 60 °C to diminish the oxides [7] (CaO) that can interfere in the silicate synthesis. For the synthesis there was calculated the mass relations considering a molar ratio of 1:1 and a batch of 1.5 g to make SCBA-sodium carbonate (Sigma Aldrich, reactant grade) pellets. The pellets were treated in a furnace (Carbolite, CWF 1300) at 800 °C and 8 hours in air atmosphere to decompose the carbonate and let react the sodium and silicon oxides.

The synthetized material was milled and analyzed by X-Ray diffraction (XRD) (Bruker D2 Phaser) to guarantee the presence of sodium silicate, then the powder was dissolved in water and methanol was added in a 2:3 water methanol ratio to generate the silanol group (Si-OH), the solution was aged for an hour to let react the silicate and the alcohol, after the hour, 3 mL of tetraethylorthosilicate (TEOS) was added and the solution was stirred two hours to generate the siloxane group (R-Si-O-Si-R) by the displacement of the H of the silanol[8]. To apply the solution over the substrates was used the spray coating method with 10, 15 and 20 wear cycles, finally the covered substrates were aged for a day before the water contact angle test.

Results and Discussion

The results from the chemical analysis of the ash are reported in Tables 1 and 2, from the elemental analysis (Table 1) is possible to demonstrate that the thermal process of calcination is able to eliminate almost 100% of the carbon in the ash, an important step considering the thermochemical synthesis method in which it is important to keep the powders compacted to let occur the reaction, if there are carbon remains in the ash, the carbon dioxide generated can damage the pellet integrity and as well the synthesis reaction.

Table 1. Elemental analysis of the SCBA.

Element	% w/w Before Calcination	% w/w After Calcination
C	15.86 ± 2.13	0.12 ± 0.05
H	0.15 ± 0.01	0
N	0.25 ± 0.05	0.09 ± 0.02
S	1.32 ± 0.09	0

From the XRF of the leached SCBA (Table 2) we obtain the inorganic composition of the ash where the silicon oxide is the main component of the SCBA followed by calcium oxide. A high concentration of CaO (no leached SCBA) can interfere in the synthesis by the formation of calcium-sodium silicates a no soluble silicate that cannot be used in the synthesis of the siloxane, nevertheless, the leached with the citric acid is insufficient to remove the necessary calcium to generate that complex silicate in a minor amount.

Table 2. XRF of leached SCBA.

Compound	%w/w Leached ash
V_2O_5	0.01
ZrO_2	0.01
Cr_2O_3	0.01
CuO	0.02
ZnO	0.03
SrO	0.05
Ag_2O	0.06
MnO	0.11
SO_3	0.25
MgO	0.29
TiO_2	0.47
P_2O_5	1.28
Al_2O_3	1.70
Fe_2O_3	4.05
K_2O	5.61
CaO	7.45
SiO_2	78.61
Total	100.00

The high content of SiO_2 makes possible to use the SCBA in the synthesis of ceramic materials and silicates as well. In Figure 1 is reported the XRD of the material after the synthesis of the sodium silicae, showing as main phase the sodium silicate and a secondary phase of sodium-calcium silicate due the CaO concentration in the ash, the low intensities are signal of a polluting phase with a low impact in the siloxane generation.

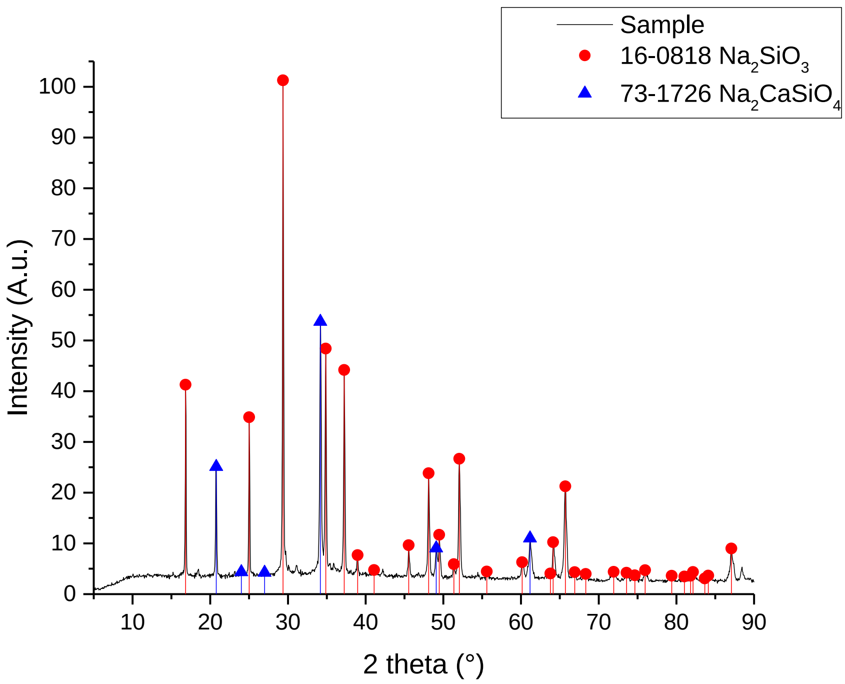

Figure 1. XRD of sodium silicate synthetized with SBCA.

The selection of the number of layers over the coated materials it´s due the generation of a white crust over the substrate, an undesirable result in this kind of coatings. To prove the coating, the surfaces selected were red clay and concrete, which were tested without coating as control and after a day of being coated, in figure 2 is possible to observe the results of the water contact angle before and after the coatings.

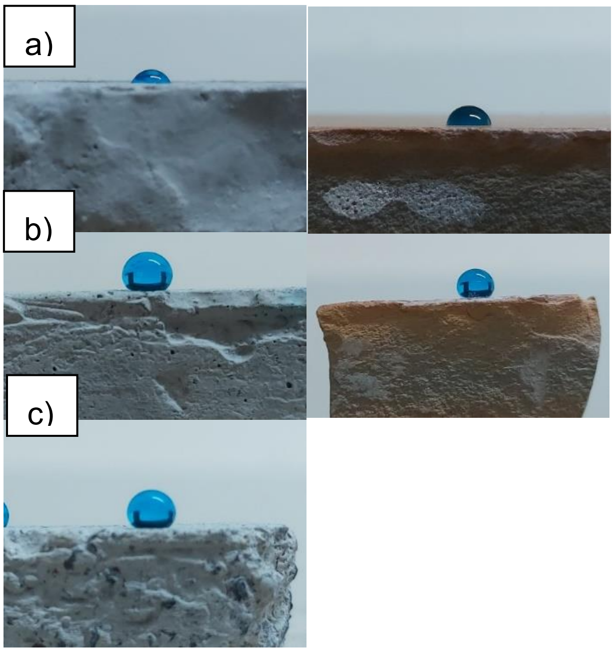

Figure 2. Water contact angle of concrete (left) and red tile (right) (**a**) without coating, (**b**) 10 layers and (**c**) 15 layers.

For the samples of red clay with 15 layers and concrete and red clay with 20 layers was impossible to quantify the water contact angle because the droplet bunces from the surface at the time it touches the material, a characteristic behavior of superhydrophobic surfaces[9]. The results of the water contact angles of figure 2 are reported in table 3.

Table 3. Water contact angle of clay and concrete.

Sample	Mean
Concrete control	71.80
10 concrete	142.10
15 concrete	146.00
Clay control	88.40
10 Clay	146.50

There are a great difference between the substrates without coating which are slightly hydrophilic with water contact angles below 90°, after being coated the materials increase the angle around 140°, a nearly superhydrophobic behavior due the interactions between the organic structures of the siloxane and the water, another important interaction occurs between the oxygen of the siloxane and the hydrogen of water, but the interaction is null compared with the covalent bond of water [3].

Conclusions

The SCBA can be used as raw materials for the synthesis of sodium silicate after being leached and calcinated to eliminate the metallic elements that can interfere in the synthesis. Although there is still a sodium-calcium silicate phase, it´s concentration is too low. This can be considered a polluting phase and does not negatively affect the coating. Sodium silicate can be used to generate coatings for ceramic materials. The samples coated showed a nearly superhydrophobic behavior and when there are 15 layers over the clay and 20 layers over the materials both showed a water repellent behavior.

References

1. W. Tang, C.I. Davidson, S. Finger, K. Vance, Erosion of limestone building surfaces caused by wind-driven rain: 1. Field measurements, Atmos. Environ. 38 (2004) 5589–5599. doi:10.1016/j.atmosenv.2004.06.030.
2. A. Erkal, D. D'Ayala, L. Sequeira, Assessment of wind-driven rain impact, related surface erosion and surface strength reduction of historic building materials, Build. Environ. 57 (2012) 336–348. doi:10.1016/j.buildenv.2012.05.004.
3. A. Venkateswara Rao, D. Haranath, Effect of methyltrimethoxysilane as a synthesis component on the hydrophobicity and some physical properties of silica aerogels, Microporous Mesoporous Mater. 30 (1999) 267–273. doi:10.1016/S1387-1811(99)00037-2.
4. D.J. Krug, R.M. Laine, Durable and Hydrophobic Organic-Inorganic Hybrid Coatings via Fluoride Rearrangement of Phenyl T12 Silsesquioxane and Siloxanes, ACS Appl. Mater. Interfaces. 9 (2017) 8378–8383. doi:10.1021/acsami.6b16121.
5. J. James, P.K. Pandian, A Short Review on the Valorisation of Sugarcane Bagasse Ash in the Manufacture of Stabilized / Sintered Earth Blocks and Tiles, 2017 (2017).
6. M.S. Sultana, M.A. Rahman, M.N. Zaman, A.N. Ahmed, Influence of Calcination on Different Properties of Sugarcane Bagasse and Waste Ash, (2015).
7. A.A. Yahya, N. Ali, N.L. Mohd Kamal, S. Shahidan, S. Beddu, M.F. Nuruddin, N. Shafiq, Reducing Heavy Metal Element from Coal Bottom Ash by Using Citric Acid Leaching Treatment, MATEC Web Conf. 103 (2017) 0–6. doi:10.1051/matecconf/201710301004.
8. U. Zulfiqar, M. Awais, S.Z. Hussain, I. Hussain, S.W. Husain, T. Subhani, Durable and self-healing superhydrophobic surfaces for building materials, Mater. Lett. 192 (2017) 56–59. doi:10.1016/j.matlet.2017.01.070.
9. C.R. Crick, I.P. Parkin, Water droplet bouncing-a definition for superhydrophobic surfaces, Chem. Commun. 47 (2011) 12059–12061. doi:10.1039/c1cc14749h.

© 2020 by the authors. Submitted for possible open access publication under the terms and conditions of the Creative Commons Attribution (CC BY) license (http://creativecommons.org/licenses/by/4.0/).