European **J**ournal of **Chem**istry

Physicochemical characterization of siliceous sands of Seme-Podji/Benin: An application for the synthesis of silica particles

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RESEARCH ARTICLE
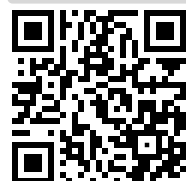
ABSTRACT

In order to valorize locally available natural resources, the silica sand in the commune of Sèmè-Podji in Republic of Benin was characterized. The results obtained by particle size, chemical analysis, X-ray diffraction (XRD), and FT-IR analysis confirmed that these sands contain a high percentage of silicon dioxide, with a value approximately equal to 90 wt.%. Low Fe_2O_3 and Al_2O_3 content (<1 wt.%) and minor oxides such as K_2O, Na_2O, TiO_2, and P_2O_5 (<0.6%) were also present. The size distributions of the particles show that the largest portion of the sand is found between 63 and 125 μm. This portion represents the one that contains the most silica. From this sand, silica particles were synthesized by the sol-gel method. The results obtained by X-ray diffraction (XRD), FT-IR analysis, and scanning electron microscopy (SEM) reveal the formation of pure amorphous silica.

Cite this: *Eur. J. Chem.* **2024**, *15(4)*, 313-319 | **Journal website**: www.eurjchem.com

1. Introduction

After air and water, sand is one of the most used resources in the world. Sand provides to the decomposition of rock near the surface of the earth. Silica sand is an industrial word used for sand which contain a very high percentage of quartz (silica) grains [1]. The most important areas of use are construction and industry. In the industry, silica sand is used in several fields whose main applications include foundry, glass industry, metal alloys, ceramics, abrasives, insulation, silicones, telecommunications, or chemicals [2]. In chemistry, silica sand is used as a raw material for porous silica synthesis [3].

Porous silica is used in biotechnology, biomedical sciences, and catalysis [4-6]. The synthesis of porous silica has been carried out by various methods using tetraethyl orthosilicate (TEOS) as a silica precursor. The most used method is the 'sol-gel' process, which has been proposed by Stöber *et al.* [7]. This method consists of hydrolysis followed by condensation of TEOS at low temperature. The high cost of this reaction has led several researchers to explore other silica precursors, such as sodium silicate [8] and other low-cost renewable natural resources such as clays and silica sand [9].

Benin has abundant deposits of silica sand that are still unexploited in the Municipality of Houéyogbé and Sèmè-Podji. The silica sand from Houéyogbé has already been characterized [2]. In this work, we are interested in the sand of Sèmè-Podji. The main objective is the valorization of Benin's siliceous sand in the synthesis of silica particles. In this study, silica sand was collected from the municipality of Sèmè-Podji and subsequently its physicochemical parameters were determined. Using this sand, pure amorphous silica particles were synthesized.

2. Experimental

2.1. Study area and sample collection

The Municipality of Sèmè-Podji, with 520 km² populated by 224,701 inhabitants [10], is located in the Department of Ouémé in the South-West of Benin at the coordinates 6°22'00''
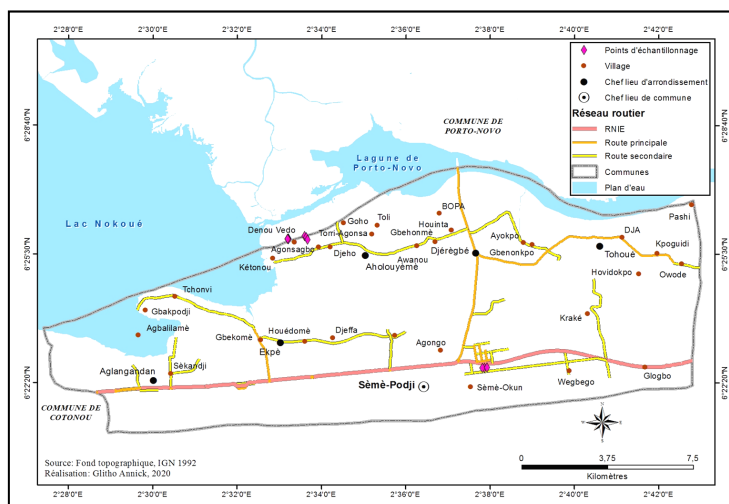
Figure 1. Municipality of Sèmè-Podji showing the sampling sites.

N and 2°37'00'' E. It is limited to the north by the cities of Porto-Novo and Aguégué, to the south by the Atlantic Ocean, to the east by the Federal Republic of Nigeria, and to the west by the city of Cotonou. This commune has six districts, including the districts of Aholouyèmè and Sèmè-Podji. The Aholouyèmè district has seven villages or city districts including Agonssagbo and Denou Vedo. The district of Sèmè-Podji has six villages or town districts, including Sèmè-Okun. The villages Agonsagbo, Denou Vedo, and Sèmè-Okun represent the places where silica sand samples were taken in each village at different points 100 m apart, using the center displacement method to obtain homogeneous portions of sand (Figure 1) [11]. In each village, three samples were collected. The samples were put into sample bags and labeled AGN1, AGN2, and AGN3 (samples collected in Agonssgbo), VED1, VED2, and VED3 (samples collected in Denou Vedo) and OKN1, OKN2, and OKN3 (samples collected in Sèmè-Okun). The sand samples are packaged separately and then brought to the laboratory.

2.2. Characterization techniques

2.2.1. pH of sand suspensions

In order to determine whether the sand can generate an acidic or alkaline aqueous solution, the pH of the suspension of samples was determined using a Mettler-Toledo pH meter. To do this, 5 g of raw sand was placed in 50 mL of distilled water and stirred for 24 hours. After this time, a pH measurement was performed on the water supernatant, after decantation of the sand suspensions [12].

2.2.2. Acid demand value (ADV)

Acid demand value (ADV) is defined as the value of acid demand of a product. When exposed to acid, elements constituting sand (such as carbonates) will react and consume the acid [2]. The higher the level of impurities, the higher the level of acid that will be consumed. This is equivalent to measuring the volume of soda needed to achieve these pH values. So, the lower ADV is the purer the sample [2]. In this method, the suspension consists of mixing 50 g of sand with 50 mL of distilled water and 50 mL of 0.1 N hydrochloric acid. The mixture of sand, water, and acid is stirred for at least one hour, then the mixture is measured with NaOH at 0.5 mol/L to determine how much of the dilute acid has reacted with the impurities in the sand. ADV was calculated corresponding to the average volume of acid consumed at pH = 3, 4, and 5 [2]. This is

equivalent to the volume of soda needed to reach these pH values.

2.2.3. X-ray diffraction

Powder X-ray diffraction (XRD) measurements were carried out at the ELETTRA synchrotron in the Materials Crystallography of X-ray diffraction (MCX) beamline, using a Huber 4-circle diffractometer with 3D translation stage and a 1D detector with a wavelength of 0.826 Å and an energy of 15 KeV. The sand samples were crushed, homogenized, and placed in 0.5 mm diameter borosilicate capillaries. The analyzes were carried out using the raw sand samples.

2.2.4. Fourier transform infrared spectroscopy

Fourier transform infrared (FTIR) measurements were performed using a Perkins Elmer Spectrum 100 device equipped with a Michelson interferometer consisting essentially of two mirrors and a semireflecting plate. The pellets used are mixtures of sample and KBr (ratio 1/150). The pellets were shaped using a hydraulic press exerting a pressure of 10 tons. The diameter of the pellets is 13 mm, and the measuring range is 4000 to 450 cm^{-1} [13].

2.2.5. Chemical analysis

The chemical analysis was carried out using a Thermo Scientific X-Ray Fluorescent Spectrometer (XRF, ARL 900 model, Measurement capacity Ppm-100%, measurement accuracy + 0.10%, with Xray tube 40 kV – 80 mA) with samples pellets prepared. Before the measurements, 10 g of material are crushed for 120 seconds then passed through the pelletizer. The obtained pellets were put in the spectrometer chamber and the measurement is carried out.

2.2.6. Particles size

The particle size analysis was carried out using a sieve (Haver & Boecker) with a diameter of 50, 63, 125, 250, 500, and 1000 μm. Six fractions are obtained at the end of each analysis.

2.3. The synthesis of silica

2.3.1. Pretreatment of sand samples for silica particle synthesis

Table 1. The acid demand value of the sand samples.

| Samples *	OKN1	OKN2	OKN3	VED1	VED2	VED3	AGN1	AGN2	AGN3
ADV (mL)	5.2	4.5	4.9	7.4	12.9	8.3	7.2	11.7	8.4

* AGN1, AGN2, and AGN3: Samples collected in Agonssgbo; VED1, VED2, and VED3: Samples collected in Denou Vedo; OKN1, OKN2, and OKN3: Samples collected in Sèmè-Okun.
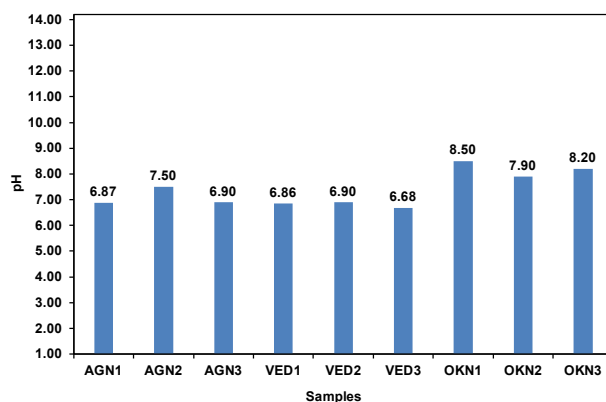
Figure 2. pH of silica sand samples.
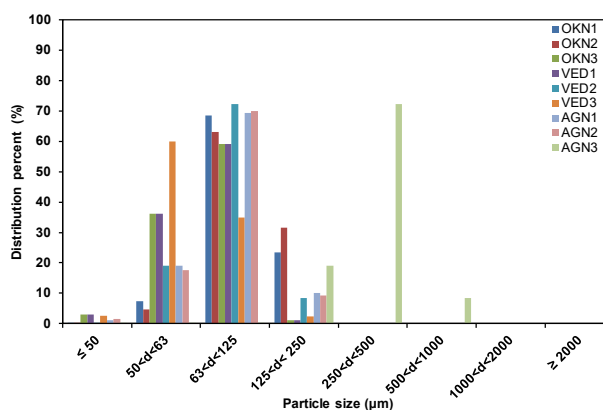
Figure 3. Sand particles size distribution.

All sand samples were sieved with a standard sieve (Manikarn) with a diameter of 250 μm. Then approximately 500 g of sand were weighed and washed three times with distilled water (centrifugation at 4500 rpm for 15 min) to remove impurities such as soluble clays. The mixture was then dried in open air at room temperature (approximately 25 °C) for 24 hours, then in an oven at 110 °C for 24 hours. The sand was finely ground with an agate mortar and resieved with 250 μm in diameter. The powder obtained was stored in plastic boxes.

2.3.2. Preparation of the precursor: Sodium silicate

The silica sand and soda pellets (NaOH) were mixed and heated for 3 hours at 450 °C in a muffle furnace. Sodium silicate is formed according to Equation 1. The obtained product was filtered to get the sodium silicate solution.

$$SiO_2 + 2\ NaOH \rightarrow Na_2SiO_3 + H_2O \qquad (1)$$
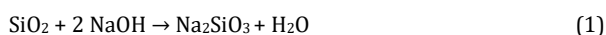
2.3.3. Synthesis of silica particles

The mixture was prepared with 50 mL of sodium silicate and 20 mL of ethanol. We then added sulfuric acid (2 M) to the sodium silicate-water mixture, with continuous stirring at 60 °C until silica gel (pH = 7), Equation 2,

$$Na_2SiO_3 + H_2SO_4 \rightarrow SiO_2 + Na_2SO_4 + H_2O \qquad (2)$$
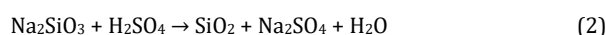
The gel obtained was left to age in the stock solution at room temperature for 24 hours. The silica gel was centrifuged at 5000 rpm several times with distilled water to separate the gel and Na_2SO_4 solution. Finally, the silica gel was dried at 60 °C for 48 hours [14]. This synthesis was also performed using potassium hydroxide as a base according to the same process.

3. Results and discussion

### 3.1. Characterization of silica sands

3.1.1. pH of sand samples

The pHs of the sand suspensions indicate whether those sands contained alkaline or acidic species. The average pH values of the sand suspensions of the silica sand samples (Figure 2) are between 6.68 and 8.50. The pH values found are around neutral pH, so the sand samples do not contain too many impurities [15].

3.1.2. Acid demand value

The acid demand value (ADV) is given in Table 1. The highest ADV value is 12.9 mL (VED2) and the lowest is 4.5 mL (OKN2).

Table 2. Chemical composition (%) of the sand samples.

Samples	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Mn_2O_3	TiO_2	P_2O_5	K_2O	Na_2O	Loss on ignition
OKN1	90.23	1.40	1.71	1.96	0.08	0.03	0.25	0.07	0.35	0.03	0.30
OKN2	88.67	1.27	1.60	2.80	0.06	0.03	0.23	0.07	0.41	0.03	0.50
OKN3	90.17	1.77	1.80	1.45	0.10	0.04	0.61	0.07	0.32	0.03	0.40
OKN Aver.	89.69	1.48	1.70	2.07	0.08	0.03	0.36	0.07	0.36	0.03	0.40
VED1	78.14	1.22	1.70	8.67	0.03	0.03	0.44	0.07	0.32	0.02	0.57
VED2	88.40	1.30	1.78	4.07	0.06	0.03	0.50	0.07	0.21	0.03	0.58
VED3	80.69	1.40	1.85	6.95	0.08	0.05	1.01	0.08	0.29	0.03	0.60
VED Aver.	82.41	1.31	1.77	6.56	0.06	0.04	0.65	0.07	0.27	0.03	0.58
AGN1	88.07	1.38	1.68	3.62	0.09	0.06	1.44	0.08	0.33	0.03	0.50
AGN2	87.51	1.34	1.76	3.87	0.07	0.05	1.04	0.08	0.26	0.03	0.80
AGN3	87.01	1.29	1.69	4.56	0.06	0.04	0.86	0.07	0.26	0.03	0.70
AGN Aver.	87.53	1.34	1.71	4.02	0.08	0.05	1.11	0.08	0.28	0.03	0.67
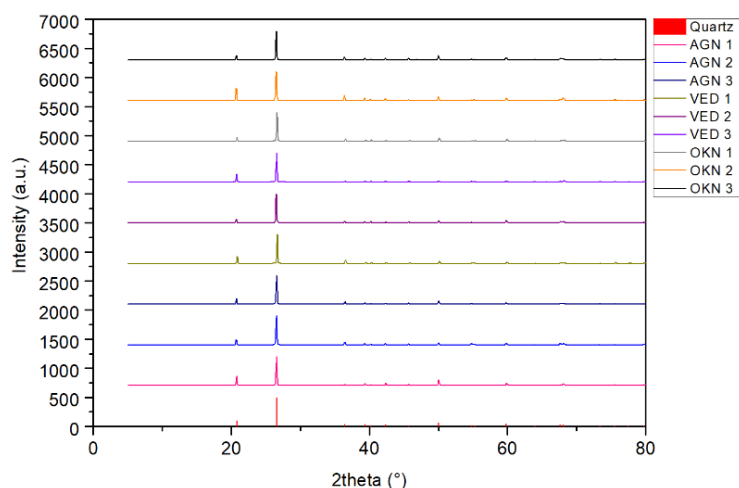
Figure 4. XRD of silica sand samples.

The acid demand value is important because it indicates the number of alkaline materials that should not be present in the washed silica sand [2]. These are minerals such as limestone and shells, $CaCO_3$, dolomite $Ca/MgCO_3$, and lime CaO [2]. The importance of acid demand in testing should not be overlooked, as it reveals various carbonates and salts that may have been missed with a standard pH test [16]. A high ADV value will significantly reduce the formation of sodium silicate and result in a decrease in the yield of the synthesized silica powder [16].

3.1.3. Particle size

The results of the particle size distributions are presented in Figure 3. The majority of samples are divided into three particle size fractions: The first particle size fraction is between 125 and 250 μm, the second size fraction is between 63 and 125 μm, and the third fraction is between 50 and 63 μm. For the AGN3 sample, the highest fraction is between 250 and 500 μm. For all other samples, the highest fractions are between 63 and 125 μm. This last fraction contains more silicon dioxide because the majority of siliceous sand grains are between 62.5 and 2000 μm [17].

3.1.4. Chemical composition

The results obtained are given in Table 2. SiO_2 percentages range from 78.14 to 90.23%. The Al_2O_3 and Fe_2O_3 oxides reached 1.77 and 1.85%, respectively. We have small amounts of potassium oxide, magnesium oxide, and sodium oxide. The loss in ignition (LOI) varies between 0.30 and 0.70%. From the results, the village of Sèmè-Okun sand samples (OKN) has the highest percentages of SiO2, with an average of around 90%. The villages of Vèdo (VED) and Agonsagbo (AGN), respectively, have an average of approximately 82.41 and 87.53% SiO_2 percentage. All silica contents of the sand samples are between

78.14% for the first sample of Vedo (VED1) and 90.23% for the first sample of Sèmè-Okun (OKN1). Therefore, they are silico-clayey sands [17]. Only the OKN1 sample was chosen to synthesize the silica particles because it has the highest silica percentage. Indeed, the silica content is essential for the formation of pure silicate, a precursor to the synthesis of silica particles.

3.1.5. X-ray diffraction (XRD)

X-ray diffraction results are shown in Figure 4. It appears that the AGN, VED and OKN samples are homogeneous and contain all quartz referenced by PDF number: 00-900-5019 (QualX2.0 Software). The characteristic peaks of alpha quartz are the most prevalent in our sand samples. In addition, all the graphs for all samples are similar.

3.1.6. Fourier transform infrared (FTIR) spectroscopy

The infrared spectra of the silica sand sample of Sèmè-Podji are shown in Figure 5. In Figure 5, all graphs are similar. Sand samples mainly contain quartz and SiO_2. Four absorption bands were located between 3000-3700 cm^{-1}, corresponding to the elongation vibrations of the internal -OH groups [18]. The band that extended between 1600-1900 cm^{-1} can be attributed to the valence vibrations of the -OH group. Furthermore, in the spectra, we have bands near 3500 cm^{-1} and a multiplet below 500 cm^{-1}. These bands were attributed to hydroxyl vibrations and iron oxide stretching, related to iron species, revealed by chemical composition investigation [19]. These species were present in sands in the form of hydroxides, as reported in the studies by Fitz Patrick *et al.* and Hongtao *et al.* [20,21].
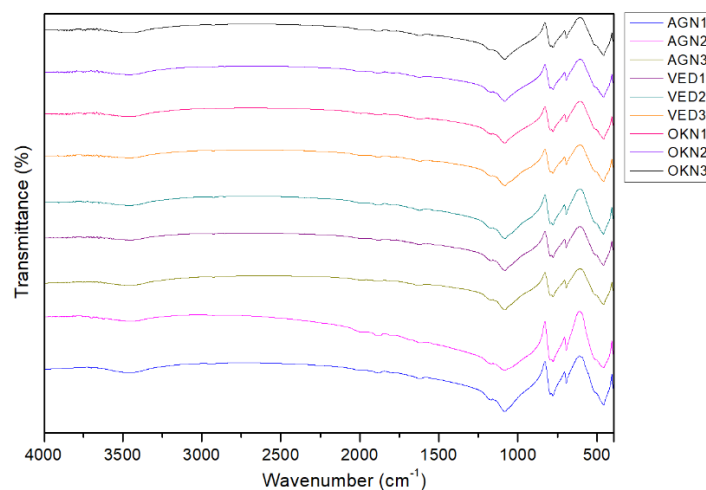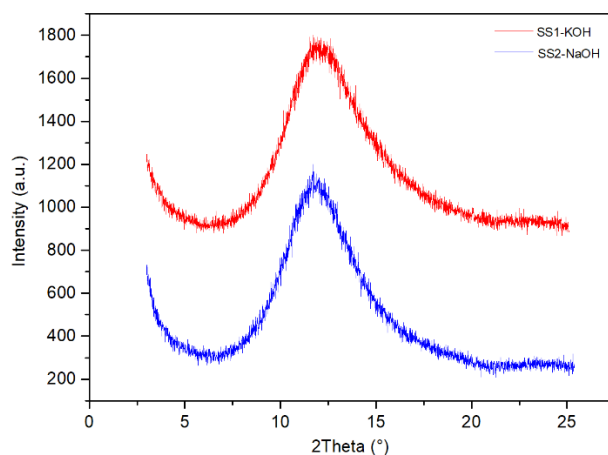
Figure 5. FTIR of silica sand samples.

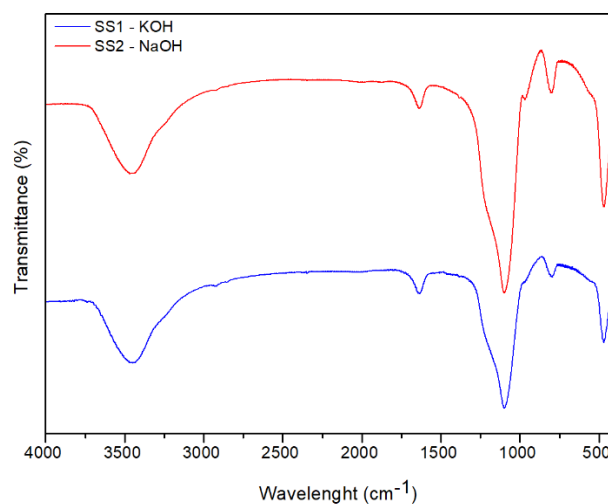**Figure 6.** XRD of the synthesized silica samples.

Figure 7. Infrared spectra of the prepared silica samples.

3.2. Characterization of the obtained silica powder

3.2.1. X-ray diffraction

Figure 6 shows the diffractograms of the synthesized silica samples. The results reveal a broad peak around 12.5° for the sample synthesized with sodium hydroxide (NaOH) and for that synthesized with potassium hydroxide (KOH). No other sharp peaks were observed in the scanning angle range, which showed the absence of any other crystal structures. This also indicated a relatively high disordered geometry of silica particles as reported by An *et al.* [22].
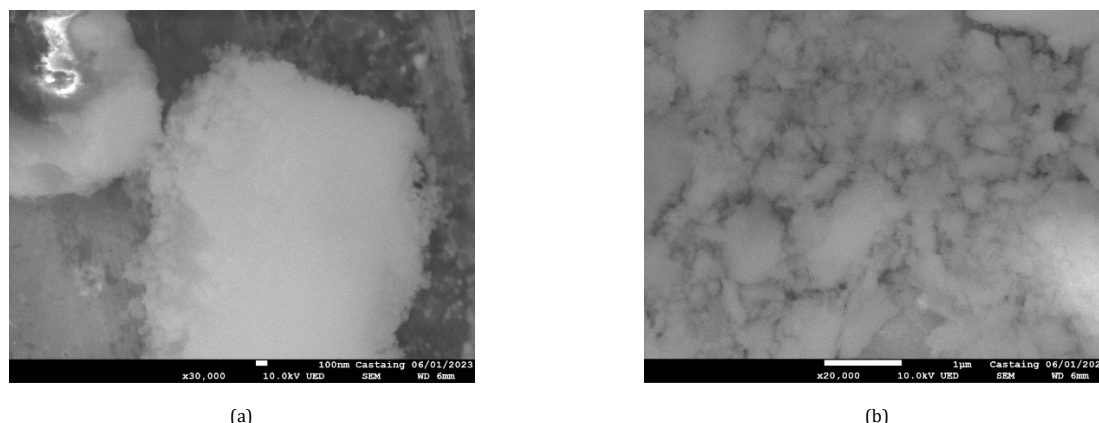
(a)

(b)

Figure 8. SEM images of the prepared silica samples (a) SS1-NaOH and (b) SS2-KOH.

According to the literature, the broad peak of amorphous silica was located around 22.5°, as shown in the results obtained by Ma *et al*. [23]. This angle is for ordinary diffractometers using Cu-radiation with a wavelength of 1.54182 Å. In our case, the analyzes were carried out at the synchrotron with a wavelength of 0.826 Å and an energy of 15 KeV, hence the shift of the silica peak to 12.5°. The sodium and potassium silicates obtained, respectively, by the action of soda and potash on silica sand were similar to those shown in the work carried out by Iler, R. K. [24]. They produced identical extrapure silica powders using this synthesis method.

3.2.2. Fourier transform infrared (FTIR) spectroscopy

Figure 7 shows the infrared spectra of silica sand samples. In Figure 7, the graphs are similar. The bands at 576, 804, and 1088 cm^{-1} correspond to the symmetric and asymmetric vibrations of the Si-O-Si group [25]. The broad band with low intensity at 3458 cm^{-1} is attributed to the stretching vibrations of the Si-OH group [25]. The band at 1634 cm^{-1} corresponds to the vibrations of water molecules (H-O-H) that are not removed during drying [25]. These results confirm the obtaining of pure silica.

3.2.3. Scanning electron microscopy (SEM)

The SEM images of the different silica samples are recorded in Figure 8. Samples SS1 and SS2 show agglomerated silica particles. This strong agglomeration is due to the drying technique used (thermal drying), as indicated by the work of Boonmee and Jarukumjorn [26]. Indeed, the formation of agglomerates is known to be excessive compared to alcoholic dehydration [26].

4. Conclusions

The results of analysis by X-ray diffraction, infrared spectroscopy, and chemical composition, the silica sand of the Sèmè-Podji analysis confirmed that these sands contain a high percentage of silicon dioxide (SiO_2), with a value approximately equal to 90 wt.%. This silica content is between 75 and 92%, characteristic of silico-clayey sands, low Fe_2O_3 and Al_2O_3 content (<1 wt.%), and minor oxides such as K_2O, Na_2O, TiO_2, and P_2O_5 (<0.6%) were also present. Beyond the appreciable results demonstrated, this silica sand was used to synthesize silica particles using the sol-Gel method using sodium silicate and potassium silicate as reaction precursors. Analysis by X-ray diffraction and infrared spectroscopy revealed the formation of pure amorphous silica. This developed material can be used as an adsorbent of pollutants or for biological applications.

Disclosure statement DS

Conflict of interest: The authors declare that they have no conflict of interest.
Ethical approval: All ethical guidelines have been adhered.
Sample availability: Samples of the compounds are available from the author.

CRediT authorship contribution statement CR

Conceptualization: Sèmiyou Ayélé Osseni; Methodology: Houefa Annick Leslie Glitho, Sourou Sidoine Bonou; Software: Lara Gigli, Jasper Plaisier; Validation: Waris Kéwouyèmi Chouti; Formal Analysis: Houefa Annick Leslie Glitho; Investigation: Houefa Annick Leslie Glitho; Resources: Etienne Sagbo, Houefa Annick Leslie Glitho; Data Curation: Lara Gigli, Asper Plaisier, Sourou Sidoine Bonou; Writing - Original Draft : Houefa Annick Leslie Glitho; Writing - Review and Editing: Houefa Annick Leslie Glitho, Sèmiyou Ayélé Osseni, Sourou Sidoine Bonou; Visualization: Waris Kéwouyèmi Chouti, Etienne Sagbo ; Supervision: Etienne Sagbo, Sèmiyou Ayélé Osseni; Project Administration: Etienne Sagbo, Sèmiyou Ayélé Osseni.

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