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Research Paper

Synthesis and Characterization of Silica from Awash Melkaasa Chemical Factory Waste

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Abstract

Silica is a common name for the chemical compound silicon dioxide. It can be found naturally in abundant amount as sand and is a raw material for many useful chemicals like silicone and silicates. It can be also found as a by-product from several chemical factories. Awash Melkassa is one of the factories that releases this silica as a waste product. In this work, this waste, that has a silica content of more than 65%, was used as a raw material for synthesis of purified silica. Here the silica was prepared by two distinct routes: basic and acidic route. In basic route, the chemical factory waste have been leached with caustic soda to prepare sodium silicate followed by reducing the silicate with an organic acid to prepare purified silica then finally size reduction by ball milling. In the second method, inorganic acids such as sulfuric acid and hydrochloric acid have been used to remove the impurities from the chemical factory waste and ball milling the purified silica to obtain the micro particles. Characterization of the final products was done by SEM and XRD. Finally the best process for silica synthesis have been decided based on which process yields the higher silica content product which is the acidic routine.

1. Introduction

Silica is another name for silicon dioxide that is the most abundant compound in the earth's crust. This compound occurs naturally as sand making up more than half of it by mass (Radhip et al., 2015). The term silica usually corresponds to geological sediment deposits rich in grains of quartz (Woronko, 2016). Besides occurring naturally, silica can be synthesised from different raw materials. This usually comes from either purification of sand (Radhip et al., 2015; Thio et al., 2020) agricultural and industrial waste (Vaibhav et al., 2015; Espíndola-Gonzalez et al., 2010) or a class of silica called bio silica which is obtained from the purification of the ashes of biomass like rice straw (Zaky et al., 2008) or husk(Moosa et al., 2017), sugar cane bagasse (Tadesse et al., 2019), coconut (Anuar et al., 2018), corn cob (Okoronkwo et al., 2016) and many more. This surge in the production of silica production is to meet the ever increasing demand for it in construction industry, cosmetic industry, chemical manufacturing industry, food industry and many more. Its inherent chemical stability and versatility in usage of many different industries like electronic device manufacturing, ceramic manufacturing, cement industry, pharmaceutical industries, food industries (packaged as powders for preservation) and so forth.(Fleming et al., n.d.; Kasaai, 2015; Bahari et al., 2011).

Among the different methods of preparing silica, the synthesis from Agricultural (biological) or industrial waste materials is becoming attractive because it is

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cheap, environmentally friendly and non-energy intensive (Singh et al., 2020). Typically, the waste from different backgrounds, which is in the form powder, is rich in silica and it will be treated with acid to remove unwanted impurities like iron oxide, aluminium oxide or calcium oxide (Kasaai, 2015). Then after, the purified powder will be leached with aqueous sodium hydroxide to form sodium silicate solution. This clear solution will be separated from other sediments by decantation or sedimentation and acids will be added to it to reduce it back to silica. The silica is then separated from the sodium salts solution formed from these acids and washed multiple times with distilled water and dried to obtain high purity silica (Zaky et al., 2008; Moosa et al., 2017).

Awash Melkassa Chemical Factory previously known as Awash Melkassa Aluminum Sulfate and Sulfuric Acid share company (AMASSASC) is a public enterprise which is located 115 kms from the capital Addis Ababa. Its main products are Sulfuric acid (H_2SO_4), aluminum sulfate ($Al_2(SO_4)_3$) and Hydrogen peroxide (H_2O_2). The aluminum sulfate sub plant uses Kaolin ($Al_2Si_2O_5(OH)_4$) a natural clay found in Hagere Selam, a place in Southern Nations, Nationalities and Peoples region and sulfuric acid to produce the alum (aluminum sulfate). In this process the acid leaches aluminum oxide from the kaolin and the residue, which is not soluble in water, will be filtered out and discarded as waste. Preparing silica from this residue is the main goal of this study.

In this study the residue named chemical factory waste (CFW) was used thermally pre-treated and used to prepare silica by two distinct routes: basic and acidic routes. Morphological characterization of the final products was done by Scanning electron microscopy (SEM) and the crystallinity of the product was analyzed by X-ray powder diffraction spectroscopy (XRD). The composition of the CFW and Kaolin was checked using X-Ray Fluorescence (XRF) spectroscopy

2. Materials and Methodology

2.1. Chemicals

The raw material is chemical factory waste (CFW) from Awash Melkassa chemical factory. Sodium hydroxide anhydrous, 99% (w/w) ALFA AESAR, Sulphuric Acid 98% (w/w) ALFA AESAR and Glacial acetic acid, 99 %(w/w) ALFA AESAR, Hydrochloric acid 35% (w/w) ALFA AESAR were used with no further purification. Distilled water was used to purify and wash the samples after every step.

2.2. Methodology

An extraction of silica from CFW was conducted by two methods: by a means of alkalization (Basic) route using NaOH and by acidic route using HCl and H_2SO_4.

A) Basic route

The alkalization route was performed under an optimal condition with the silica to NaOH ratio of 1:1.33 on dry basis (w/w). First the chemical waste was fetched from Awash Melkassa chemical factory and the amount of waste that we need for our project was weighed out. The CFW was treated by heat in the furnace for 4 hours at 700°C, and then it was milled by a ball milling for 10 minutes at 500 RPM. After that the silica extraction process was started. To prepare sodium silicate (Na_2SiO_3) 43.6 g the CFW was reacted with an alkaline solution by dissolving 38.8 g sodium hydroxide (NaOH) in 500 ml of distilled water. This process has been carried out at 90°C for about three hours.

Afterwards, the solution was stirred for about 4 hours at 25°C using magnetic stirrer. After this process had been completed the residue was filtered out from silicate solution by using vacuum pump. Then acetic acid was added to the sodium silicate solution to reduce the sodium silicate in to silica or silicon dioxide. In this step an excess amount of acetic acid was to the aqueous solution to reduce all of the sodium silicate. The result was formation of white residue, sodium acetate and water. The white residue is silicic acid which is the hydrated form of silica.

This residue and liquid was separated using vacuum filtration process. Then, the white residue was washed with water repeatedly to remove the impurities until the pH reached seven. Since silica is not soluble in water it cannot be affected by water addition. But, the impurity can be removed with water. Afterwards, the white residue was dried in an oven for 3 hours at 90°C

Finally, the synthesized silica was ground in the ball mill to produce a fine powder and characterization was carried out. The reaction process that may occur is the following:

$$CFW + 2NaOH_{aq} \rightarrow Na_2SiO_{3aq} + H_2O$$

$$Na_2SiO_3 + CH_3COOH \rightarrow SiO_2 + Na_2CH_3COO + H_2O$$

B) Acidic route

The second method of extraction that had been used to extract silica from CFW was acidic route. In the acidic route HCl and H_2SO_4 was being used in the extraction process. In this step, HCl and H_2SO_4 was being added in to the CFW and the process was carried out for 2 hours at 100 ^{0}C. This process aimed to prepare silica by removing the impurities of the chemical factory waste like alumina and iron oxide with the help of acid to convert them into salts that can be washed away analysis.

3. Result and Discussion

The Awash Melkassa chemical factory releases the CFW as a waste product at the end of the production of aluminium sulphate. The Figure 1 shows CFW of Awash Melkassa chemical factory. Since it is a residue of kaolin which is greyish-white powder, the CFW is also a grey powder. After the calcination, this CFW is completely white. This may be due to removal of oxide contaminants and the complete evaporation of water from it.

Figure 1: Chemical factory waste (left) and Kaolin deposit (right)

The chemical composition of the kaolin is shown in Table 1.The composition analysis was done using X-Ray Fluorescence (XRF) spectroscopy. Although kaolin is primarily used in the cement and construction industry, it can also be used as a raw material to produce alum (aluminium sulphate) (Ojewumi et al., 2018) and silica (Rahayu et al., 2018).

The CFW had similar chemical compositions compared to that of the kaolin. The only difference was of the amount of the major components silica and alumina that can be seen in the Table 2.

The increment of silica and the decrement of alumina values can be explained by the acid leaching of the kaolin. Awash Melkassa chemical factory prepares alum from kaolin by leaching most of the alumina out of it with sulphuric acid at high temperature and pressure (162°C, 4 bar).The alum is dissolved why the residue (CFW) is discarded.

Table 1: Chemical composition of Ethiopian Kaolin

Chemical compound	Composition (Weight %)
SiO_2	57.633
Al_2O_3	37.766
Fe2O3	0.86
MgO	0.596
CaO	0.346
K_2O	1.801
TiO_2	0.605
P_2O_5	0.311

Table 2: Chemical composition of the CFW

Chemical compound	Chemical composition (Weight %)
SiO_2	68.633
Al_2O_3	17.766
Fe2O3	0.86
MgO	0.596
CaO	0.346
K_2O	1.801
TiO_2	0.605
P_2O_5	0.311

The amount and the quality (absence of impurity) of silica found using acidic route was better, albeit not significantly, than that of Basic route. The smaller of silica may be due to loss of silica as sodium silicate in filtration step in the basic route and the loss in quality may be due to presence of metal oxide impurity or possible formation of sodium aluminate as a by-product in the basic route which is an impurity latched on the surface of the silica.

The morphology of the basic silica (BS) powder was investigated using scanning electron microscopy. The SEM images reviled an irregular shaped agglomeration of the micro particles, when viewed around 25,000 X magnification in Figure 3b. The particle size was measured using image J-software. It was found that the particles had an average size of 112 µm. The agglomeration of the particles may be due to the impurities found in the sample. This may have risen from the deposition of unwashed sodium acetate or sodium aluminate on the particles or impurities from planetary ball mill which cannot be removed even after repeated washing.

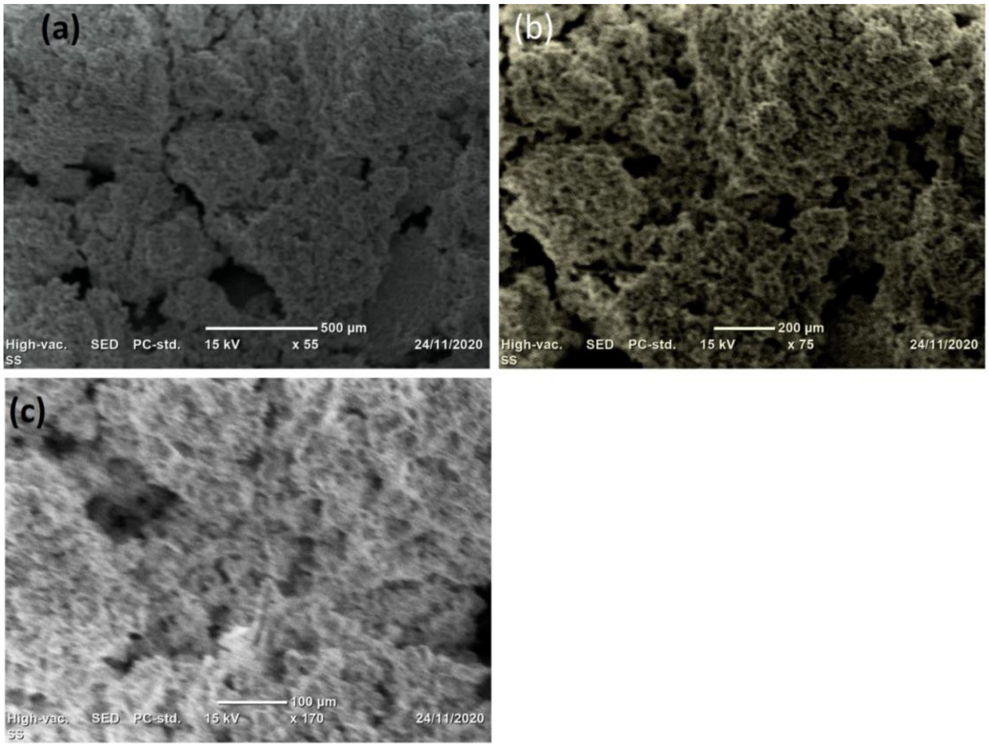

Figure 3: The SEM images of silica micro particles in different resolution of (a) 500 µm, (b) 200 µm, and (c) 100 µm.

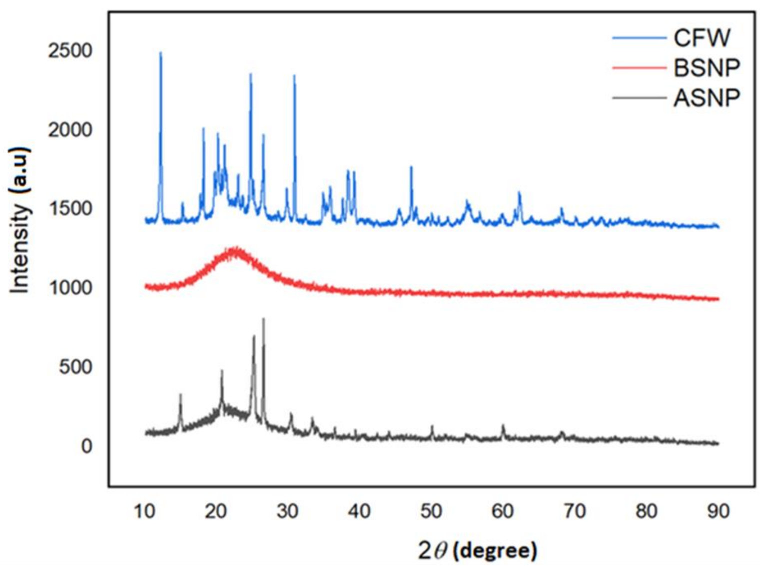

Figure 4: XRD analysis of three samples, chemical factory waste (CFW), basic step silica microparticles (BSNP) and acidic step silica microparticles (ASNP).

Figure 4 shows XRD pattern of the silica particles, the sharp peaks at the angles below 20o might indicate the crystalline phases of alumina in the CFW. This can be further observed by their disappearance in the acidic silica micro particles. Sharp spectrum appearing at 2θ = 22, might indicates the crystalline phases of silica present in both the CFW and Acidic route silica novel particles (ASNP). The disappearance of the sharp peaks and the softness of the band at the Basic route silica novel particles BSNP might indicate the presence possible impurities and the amorphous nature of the silica micro particles prepared in the basic route. Amorphous impurity in the crystalline material may cause shift in the peaks of XRD pattern and change in intensity of peaks. There may be change in bond length and bond stretching. The XRD compatibility test done on a software shows the samples are most probably kaolin and silica.

4. Conclusion

In this work silica was synthesized in 2 methods, named acidic and basic route. The synthesized silica was characterized using SEM and XRD. Using the acidic routine silica was synthesized successfully with accuracy of 99% match and this was found with the aid of XRD. 20 μm, 50 μm, 100 μm, 200 μm and 500 μm particle size of silica was produced, using a planetary ball miller. In both routes some agglomeration had been seen on the particles this result is found from XRD and SEM. From this, it can be concluded that there is small amount of impurities in the product that leads to agglomeration of the product. This may be due to the adhesion of impurities like iron oxide and alumina into the chemical structure of silica. Besides that the acidic route showed better quality of silica in terms of color smaller amount of impurity deduced from the XRD analysis. This shows the fact that silica can be prepared from chemical factory waste and leaching with acid gives better yield.

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