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Production of High-Purity Silica Sand from Ivorian Sedimentary Basin by Attrition without Acid Leaching Process for Windows Glass Making

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Abstract

To produce high-purity silica sand usable for glass making, the present study was carried out. The objective of this work was to increase the silicon dioxide (SiO_2) content to at least 99% using a simple process without chemical input. The raw sand samples were taken from the Ivorian sedimentary basin, from Maféré and Assinie areas, Côte d'Ivoire. Wet sieving and attrition technique were used for the purification process. The results from the energy dispersive spectrometer (EDS) analyses of the raw and treated samples show a significant increase of silica content and a significant reduction of impurities. The silica content (SiO_2) of the sand of Maféré increases from 98.73% ± 0.15% to 99.92% ± 0.05%. And the sand of Assinie increased from 98.82% ± 0.67% in the raw samples to 99.44% ± 0.27% after treatment. The rate of iron oxide and alumina is reduced in these sands. Moreover, the sand of Maféré contains 53.2% of grains of size lower than 500 microns and that of Assinie contains 29.30%. Regarding the chemical composition of these purified sands, they meet the standard BS2975s, the American Ceramic Society and the National Bureau of Standards for window glass making.

Keywords

Silica Sand, Silicon Oxide, Attrition, Wet Sieving, Soda Lime Glass, Maféré, Assinie, Côte d'Ivoire

1. Introduction

Window glass making requires the use of very pure silica sand without impurities [1]. Other domains such as photovoltaic cell production, silicon metal wafers, and optical glass are also dependent on good quality silica [2] [3] [4] [5] [6]. The BS2975 (British Standards International) specification published in 2008 specifies the silicon oxide (silica) content and the limits of iron, chromium, and titanium oxides in good quality silica sands. However, natural silica can be used if it meets the quality requirements of the standards [7] [8] [9]. According to this standard, there are three distinct grades of sand named A, B and C. Grade A contains a maximum of 0.008% Fe_2O_3, 0.030% TiO_2, 2 ppm Cr_2O_3 and a minimum of 99.5% silica, Grade B sand contains a minimum of 99.5% silicon oxide and a maximum of 0.013% Fe_2O_3 and 2 ppm Cr_2O_3, while Grade C contains a minimum of 98.5% SiO_2, a maximum of 0.035% Fe_2O_3 and 6 ppm Cr_2O_3 [9]. Grade A sand is used for the manufacture of optical glass, Grade B sand is used for the manufacture of household objects and colored glass. Grade C sand, suitable for the manufacture of color less containers etc., should have a maximum Fe_2O_3 content of 0.030% and not more than 6 ppm Cr_2O_3. There is a proviso that the Fe_2O_3 specification can be relaxed to 0.035% maximum if the sand contains less than 2 ppm Cr_2O_3. A minimum SiO_2 content of 98.5% is specified.

Previous work has led to the discovery of silica sand (S.S) with a high silica content in the Ivorian sedimentary basin [10]. The sands with a high proportion of silicon oxide (silica) are found east of Abidjan up to the border with Ghana. In the locality of Maféré, Aboisso region, Côte d'Ivoire, siliceous sands are found with an average silica content of 97.41%. These sands are fine with 66.8% of the grains less than or equal to 500 microns in size. The quality of these sands is suitable to produce silica glass [10].

However, pure sand with a silica content of 98% or more is required to produce window glass. Highly pure silica sands with a silica content of more than 99.00% are required to produce optical glass and for the manufacture of photovoltaic cells. In this perspective, this study was carried out to propose a method of simple treatment, without the use of chemical products on the said sands. Thus, after the sampling phase, treatment of the samples was carried out in the laboratory using only wet sieving and attrition.

2. Material and Methods

2.1. Collection and Conservation of Samples

The sampling phase was done according to the method described in our previous work [10] [11]. Samples were taken with an auger at a depth of 50 centimeters. Composite sand samples $(S.S)_0$ of 1050 grams were collected in Maféré and Assinie, Côte d'Ivoire. **Figure 1** illustrates the location of the sampling sites. The samples were transported to the laboratory in canvas bags free of any contamination at room temperature.

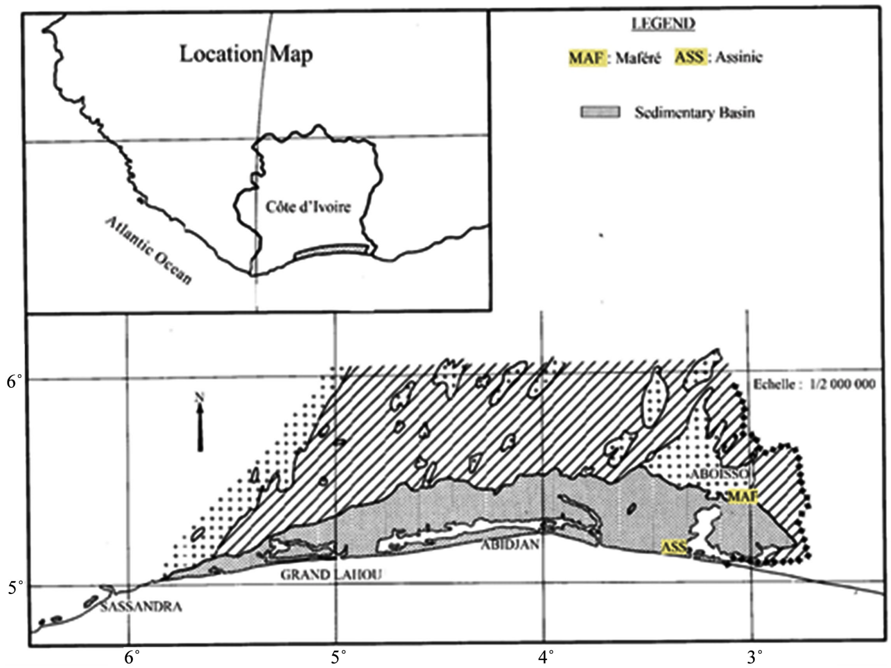


Figure 1. Sand sample collection map ([12] amended).

2.2. Sample Preparation for the Analysis Phase

In the laboratory, a fraction of 750 grams of each sample $(S.S)_0$ was dried. The drying was done at 105 degrees Celsius to constant mass. After cooling in the desiccator, a weight of 50 gram of each raw sample was pulverized (to a 75 micrometers particle size) for chemical composition analysis by energy dispersive spectrometer (EDS). Thereafter, 500 grams of each dried sample was used for particle size analysis. Finally, 200 grams of the raw samples $(S.S)_0$ was utilized for the processing phase.

2.3. Wet Sieving and Attrition Technique

The processing procedure begins with wet sieving of 200 g of the raw samples $(S.S)_0$ with distilled water using a one-millimeter mesh size for removal of coarse particles and debris. The passing at this mesh is sifted, still in wet way, to 80 microns for the elimination of the fine clayey fraction and limestones. Thereafter, with the refusal of the sieve to 80 microns the attrition is carried out using the device of **Figure 2**. The equipment consists of a plastic test tube and an electric agitator used to shake the contents of the test tubes.

Attrition is the most common enrichment method for sand enrichment [13] [14] [15] [16]. In this study, we introduce the 80 microns refusals of each sample into test tubes. Then, 300 milliliters of distilled water were added, and the test tubes were closed tightly. Finally, the mixture of sand and distilled water was agitated by a stirring machine that performs an oscillatory movement (180 oscillations per minute), with an amplitude of 20 centimeters. This movement creates friction between the sand grains.

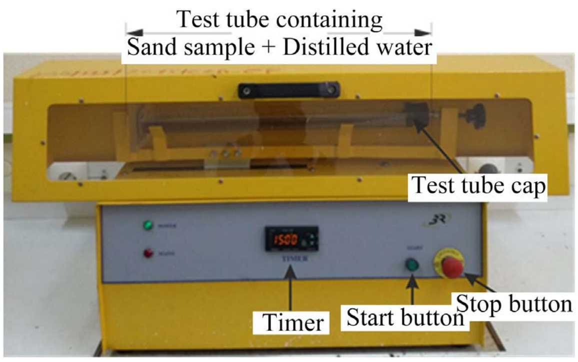


Figure 2. Device used for sample attrition.

The attrition was carried out in two cycles for 15 minutes each [14] [17]. At the end of each 15-minute attrition sequence, the contents of the attrition vessel are sieved to 80 microns with distilled water.

The nomenclature of sand samples adopted in this manuscript is as follows: $(S.S)_{(i,j)}$ with, $i = \{MAF; ASS\}$: sampling sites and $j \in [\![0;1]\!]$: processing level. The Table 1 is the summary of the sample nomenclature.

The product of the last sieving carried out is oven dried. The schematic of the processing protocol is shown in Figure 3.

Finally, a 50-grams fraction of the treated and dried sands is ground for chemical composition analysis.

2.4. Analysis of the Samples

Several analyses were carried out on the sand samples. First, the chemical composition of the raw and treated sands was analyzed using the energy dispersive spectrometer (EDS). Then, the granularity of the raw sands was determined. Finally, the micrography of the quartz grains is performed by Scanning Electron Microscope (SEM).

Energy Dispersive Spectrometer (EDS)

Energy dispersive spectroscopy (EDS) provides elemental and chemical analysis of samples [18] [19]. We use the crushed material of each sample (raw and processed) for the determination of the chemical composition. The principle of this analysis is to first ionize the atoms of the samples. Then the instrument measures the intensity and energy of the X-rays emitted by the atoms. The results of this method are the chemical composition and emission spectra of each sample.

Granulometric analysis

A weight of 500 grams of each previously dried crude was used. A series of AFNOR sieves (80 µm, 100 µm, 125 µm, 160 µm, 200 µm, 250 µm, 315 µm, 400 µm, 500 µm, 630 µm, 800 µm, 1 mm, 1 mm. 25 mm, 1.6 mm, 2 mm, 2.5 mm) are used. The column is placed on a vibrating sieve machine for 10 minutes. Each sieve reject is weighed for determination of the proportions of pass and reject [11].

Table 1. Nomenclature of raw and processed samples.

Designation	Raw samples	Processed samples
MAF: Maféré	$(S.S)_{MAF,0}$	$(S.S)_{MAF,1}$
ASS: Assinie	$(S.S)_{ASS,0}$	$(S.S)_{ASS,1}$

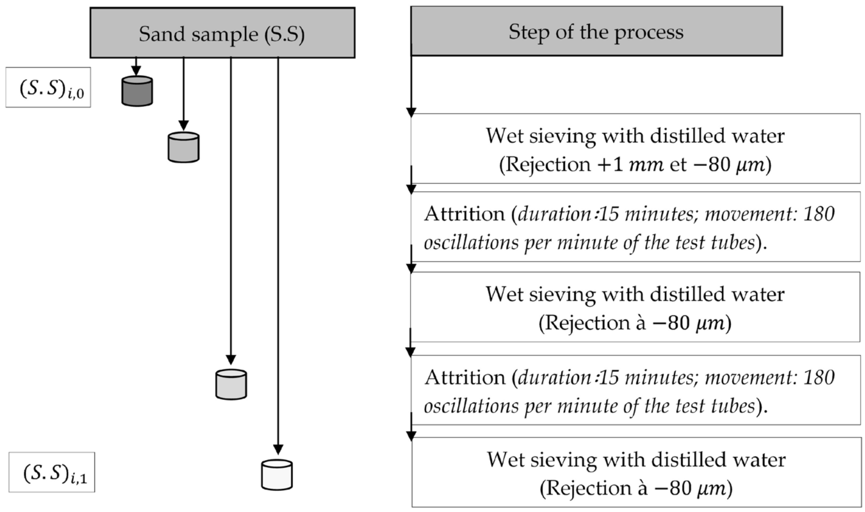


Figure 3. Scheme followed for the purification of silica sand samples.

Data from the particle size analysis are used for sand characterization. We use the method of statistical moments and that of Folk and Ward. These methods are described in the article by Blott and Pye [20]. They proposed a macro running under Microsoft Excel© (GRADISTATv9.1xlsm) for the characterization of samples based on the particle size analysis. The good grading of the sands is also evaluated using the indices from the method of moments and the Folk and Ward method. These are parameters such as: means, mode(s), standard deviation, skewness, kurtosis, and a range of values of cumulative percentiles (the grain size at which a specified percentage of the grains are coarser), namely D_{10}, D_{50}, D_{90}.

The particle size scale used was proposed by Blott [11] [20]. Table 2 illustrates the nomenclature of grain size classes. Grain sizes are expressed in mm (x_{mm}) or µm ($x_{µm}$) but also in phi (ϕ). The relationship between these units' systems is established by the following expression:

$$x_{\phi} = -\log_2(x_{mm}).$$

Processing weight efficiency

Next, we evaluate the yield (η) of the processing method for raw sand samples.

The adopted calculation method is illustrated by the formulas in Table 3 and in Table 4. We use the results of the spectrometric analysis to calculate the different yields.

These formulas, Table 3, and Table 4 consider the objective of silica sand treatment, which is to increase the silica content to 100% by removing impurities [21]. Indeed, a treatment method would be efficient (ideal) if it allows the reduction of impurities to 0% and raises the silica content to 100%. Thus, we calculated the yield on the silica content by the ratio of the difference between the final proportion (example $T_{(silica)MAF,1}$) and the initial proportion (example $T_{(silica)MAF,0}$) and the difference between 100% and the initial proportion (example $T_{(silica)MAF,0}$). For the impurities, the calculation of the yield was using the difference between 0% and the initial proportion (example $T_{(silica)MAF,0}$) in the denominator.

Scanning Electron Microscope (SEM)

Exoscopy allows us to observe and interpret this surface condition by analyzing images taken by scanning electron microscopy (SEM), at magnifications typically between ×500 and ×20,000. Nearly 250 characters have been listed [22] [23]. They allow to determine the depositional environment of a grain, its history and sometimes its geographical origin. It is strongly recommended that the 250 - 355 µm fraction be preferred [11].

Table 2. Size scale proposed by Blott.

Grain size		Descriptive terminology	
phi	mm/µm		
−6 to −5	32mm - 64 mm	Very coarse	Gravel
−5 to −4	16 - 32	coarse	
−4 to −3	8 - 16	Medium	
−3 to −2	4 - 8	Fine	
−2 to −1	2 - 4	Very fine	
−1 to 0	1 - 2	Very coarse	Sand
0 - 1	500 µm - 1 mm	Coarse	
1 - 2	250 µm - 500 µm	Medium	
2 - 3	125 - 250	Fine	
3 - 4	63 - 125	Very fine	

Table 3. Calculation of the yield of the treatment performed for Sand sample of Maféré.

Compound	yield of the treatment performed (η)
	Sand sample of Maféré
Silica	$\eta_{(silica)} = [T_{(silica)MAF,1} - T_{(silica)MAF,0}]/[100 - T_{(silica)MAF,0}] \times 100$
Iron oxide	$\eta_{(iron)} = [T_{(iron)MAF,1} - T_{(iron)MAF,0}]/[0 - T_{(iron)MAF,0}] \times 100$
Alumina	$\eta_{(alumina)} = [T_{(alumina)MAF,1} - T_{(alumina)MAF,0}]/[0 - T_{(alumina)MAF,0}] \times 100$

Table 4. Calculation of the yield of the treatment performed for Sand sample of Assinie.

Compound	yield of the treatment performed (η)
	Sand sample of Maféré
Silica	$\eta_{(silica)} = [T_{(silica)ASS,1} - T_{(silica)ASS,0}]/[100 - T_{(silica)ASS,0}] \times 100$
Iron oxide	$\eta_{(iron)} = [T_{(iron)ASS,1} - T_{(iron)ASS,0}]/[0 - T_{(iron)ASS,0}] \times 100$
Alumina	$\eta_{(alumina)} = [T_{(alumina)ASS,1} - T_{(alumina)ASS,0}]/[0 - T_{(alumina)ASS,0}] \times 100$

3. Results and Discussion

At the end of the implementation and the different analyses carried out on the sand samples, some results were found.

3.1. Particle Size Analysis

Glass production requires sand with a fine grain size of about 200 microns to 500 microns [13] [14]. Figure 4 and Figure 5 represent the graphical results of the particle size analysis of the sands of Maféré and Assinie respectively. These figures show the proportions of passing on the sieves of the AFNOR series (from 80 microns to 2.5 millimeters).

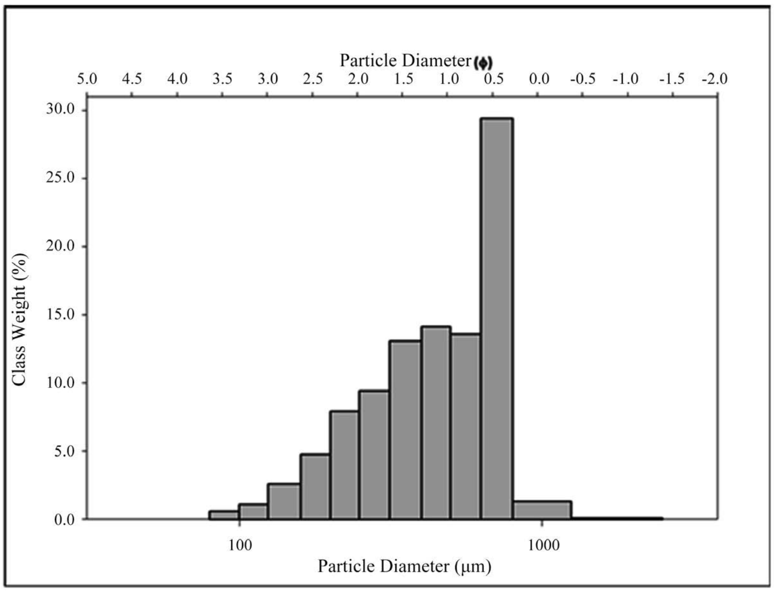


Figure 4. Grain size distribution of Maféré Sand.

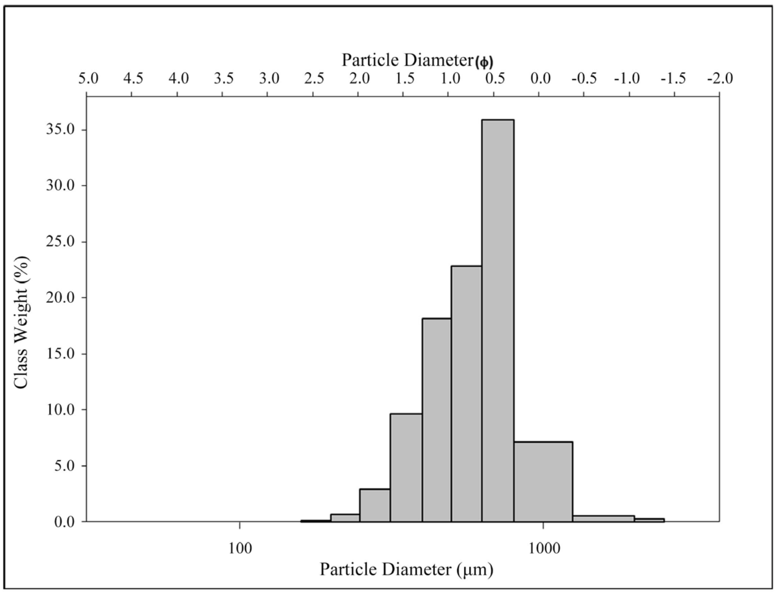


Figure 5. Grain size distribution of Assinie Sand.

The grain size distribution of Maféré sand is bimodal with one modal value at 450 micrometers and the other at 715 micrometers (respectively ϕ = 0.494 and ϕ = 1.61) (see Figure 4 and Table 5). Other position indices such as D_{10} and D_{90} are equal to 206 (ϕ = 0.403) micrometers and 756 micrometers (ϕ = 2.278) respectively.

Also, in the Sand-Clay-Silt diagram, the Maféré sample is a sand containing 53.2% of grains smaller than 500 microns (Figure 6). There are 26.6% of grains between 250 microns and 500 microns in this sand.

Furthermore, according to the indices calculated by the method of moments and by the method of Folk and Ward (see Table 6), the sand of Maféré is a medium sand, moderately sorted. The fine granularity of the sand up to 125 microns is suitable for glass production [10] [13].

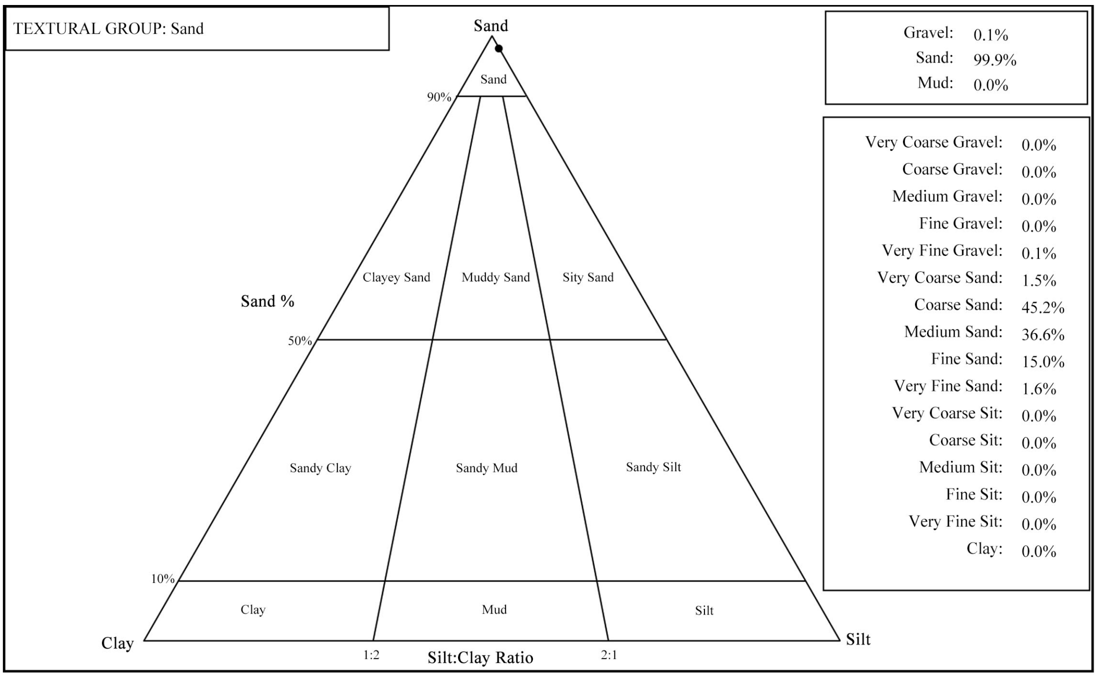


Figure 6. Maféré sand in the ternary Sand-Clay-Silt diagram.

Table 5. Statistical parameters of position of Maféré sand and Assinie Sand.

Sample	D_{10}	Median ($D5_0$)	Mode(s)	D_{90}
$(S.S)_{MAF,1}$	206 µm	474 µm	450 µm and 715 µm	756 µm
$(S.S)_{ASS,1}$	372 µm	624 µm	715 µm	924 µm

Table 6. Parameters of the method of moments and the method of Folk and Ward of Maféré sand.

Parameters	Method of moments			Folk and Ward's method		Description
	Arithmetic (µm)	Geometric (µm)	Logarithmic phi (ϕ)	Geometric (µm)	Logarithmic phi (ϕ)	
Mean	492.663	434.026	1.204	438.087	1.191	Medium Sand
Sorting	225.084	1.676	0.745	1.658	0.730	Moderately Sorted
Skewness	0.726	−0.645	0.645	−0.288	0.288	Fine Skewed
Kurtosis	6.062	2.845	2.845	0.817	0.817	Platykurtic

In the case of the Assinie sample, its particle size distribution is unimodal. The modal value is 715 micrometers (respectively $\phi = 0.494$) (see Figure 7 and Table 7). Other positional indices such as D_{10} and D_{90} are equal to 372 ($\phi = 0.114$) micrometers and 924 micrometers ($\phi = 1.425$) respectively.

In the Sand-Clay-Silt diagram, the Assinie sample is a sand containing 29.3% of grains smaller than 500 microns. The proportion of fine phase is very low. There are 0.7% of grains between 250 microns and 500 microns in this sand (Figure 7).

Moreover, regarding the indices calculated by the method of moments and by the method of Folk and Ward, the sand of Assinie is well sorted around the average diameter. The characteristic curve of its histogram is of mesokurtic form. This sand is coarser than that of Maféré (see Table 4).

Thus, the sand from Maféré would be more useful for glass production than that from Assinie. In addition to the granularity of these sands, we are interested in the chemical composition of these sands.

Table 7. Parameters of the method of moments and the method of Folk and Ward of Assinie sand.

Parameters	Method of moments			Folk and Ward's method		Description
	Arithmetic (μm)	Geometric (μm)	Logarithmic phi (ϕ)	Geometric (μm)	Logarithmic phi (ϕ)	
Mean	642.7	598.7	0.740	590.8	0.759	Coarse Sand
Sorting	235.5	1.415	0.501	1.408	0.494	Well Sorted
Skewness	1.524	−0.090	0.090	−0.164	0.164	Fine Skewed
Kurtosis	8.766	3.372	3.372	1.089	1.089	Mesokurtic

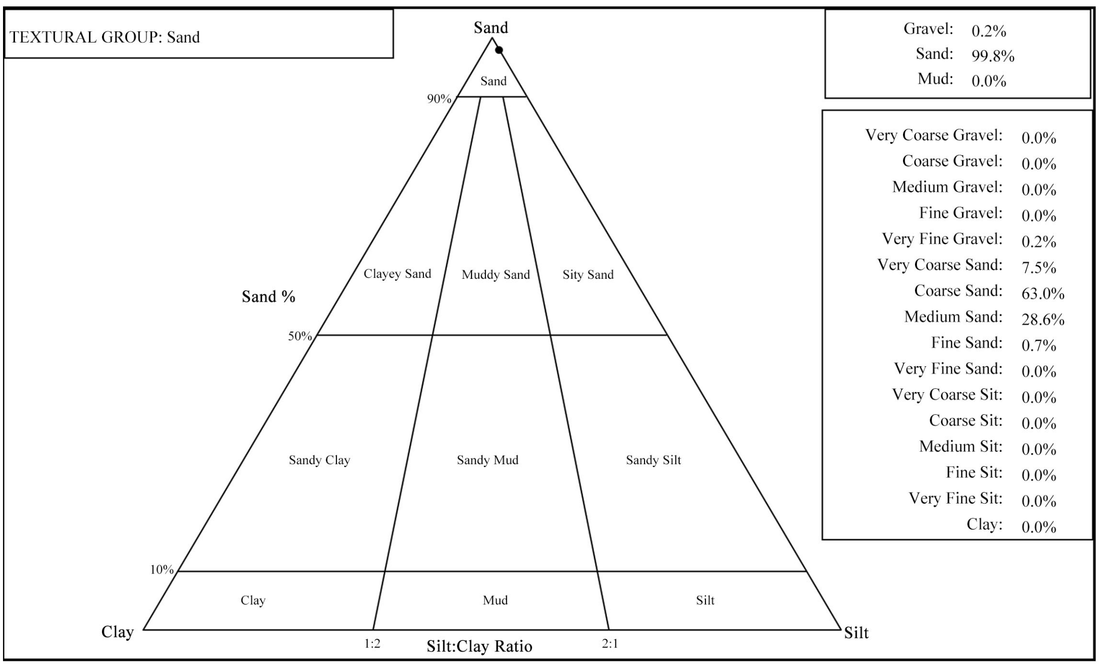


Figure 7. Assinie sand in the Sand-Clay-Silt ternary diagram.

3.2. Chemical Composition of Sedimentary Basin Sand

The results of the chemical composition analysis of the samples are shown in Table 8 and Table 9. Three analyses were performed for each sample.

Table 8. Chemical composition of raw and treated sands from Maféré.

$(S.S)_{MAF,0}$			
Parameters	Chemical compound		
	Al_2O_3 (%)	FeO/Fe_2O_3 (%)	SiO_2 (%)
sample 1	0.18	0.99	98.83
sample 2	0.24	0.97	98.79
sample 3	0.59	0.86	98.56
Mean	0.33	0.94	98.73
Std. deviation	0.22	0.07	0.15
$(S.S)_{MAF,1}$			
	Al_2O_3 (%)	FeO/Fe_2O_3 (%)	SiO_2 (%)
Sample 1	0.09	0.03	99.88
sample 2	0.09	0.01	99.90
sample 3	0.00	0.03	99.97
Mean	0.06	0.03	99.92
Std. deviation	0.06	0.01	0.05

Table 9. Chemical composition of raw and treated sands from Assinie.

$(S.S)_{ASS,0}$			
Parameters	Chemical compound		
	Al_2O_3 (%)	FeO/Fe_2O_3 (%)	SiO_2 (%)
sample 1	1.40	0.19	98.41
sample 2	0.17	0.24	99.59
sample 3	1.28	0.26	98.46
Mean	0.95	0.23	98.82
Std. deviation	0.68	0.04	0.67
$(S.S)_{ASS,1}$			
	Al_2O_3 (%)	FeO/Fe_2O_3 (%)	SiO_2 (%)
Sample 1	0.87	0.00	98.94
sample 2	0.22	0.12	99.53
sample 3	0.22	0.26	99.61
Mean	0.44	0.13	99.44
Std. deviation	0.37	0.13	0.27

Energy dispersive spectrometry revealed that the raw sand from Maféré contains 98.73% ± 0.15% silica. This sand also contains 0.94% ± 0.07% iron oxide and 0.33% ± 0.22% alumina. After treatment, the chemical composition of this sand changes. The proportion of mineral silica increases to 99.92% ± 0.05%. The content of other oxides is reduced. The iron oxide remains at 0.03% ± 0.01% in this sand. And there remains 0.06% ± 0.06% of alumina (see Table 8).

Moreover, the raw sand of Assinie contains 98.82% ± 0.67% silica. This sand also contains 0.23% ± 0.04% iron oxide and 0.95% ± 0.68% alumina. After processing, the chemical composition of this sand also changes. The proportion of mineral silica increases to 99.44% ± 0.27%. The content of other oxides was reduced while the iron oxide remains at 0.13% ± 0.13% and alumina at 0.44% ± 0.37% (see Table 9).

The chemical compositions revealed by energy dispersive spectrometry are illustrated by the spectra in Figure 8 and Figure 9. These Figures showed the energy spectra of the six EDS analyses carried out on the sand of Maféré and Assinie respectively.

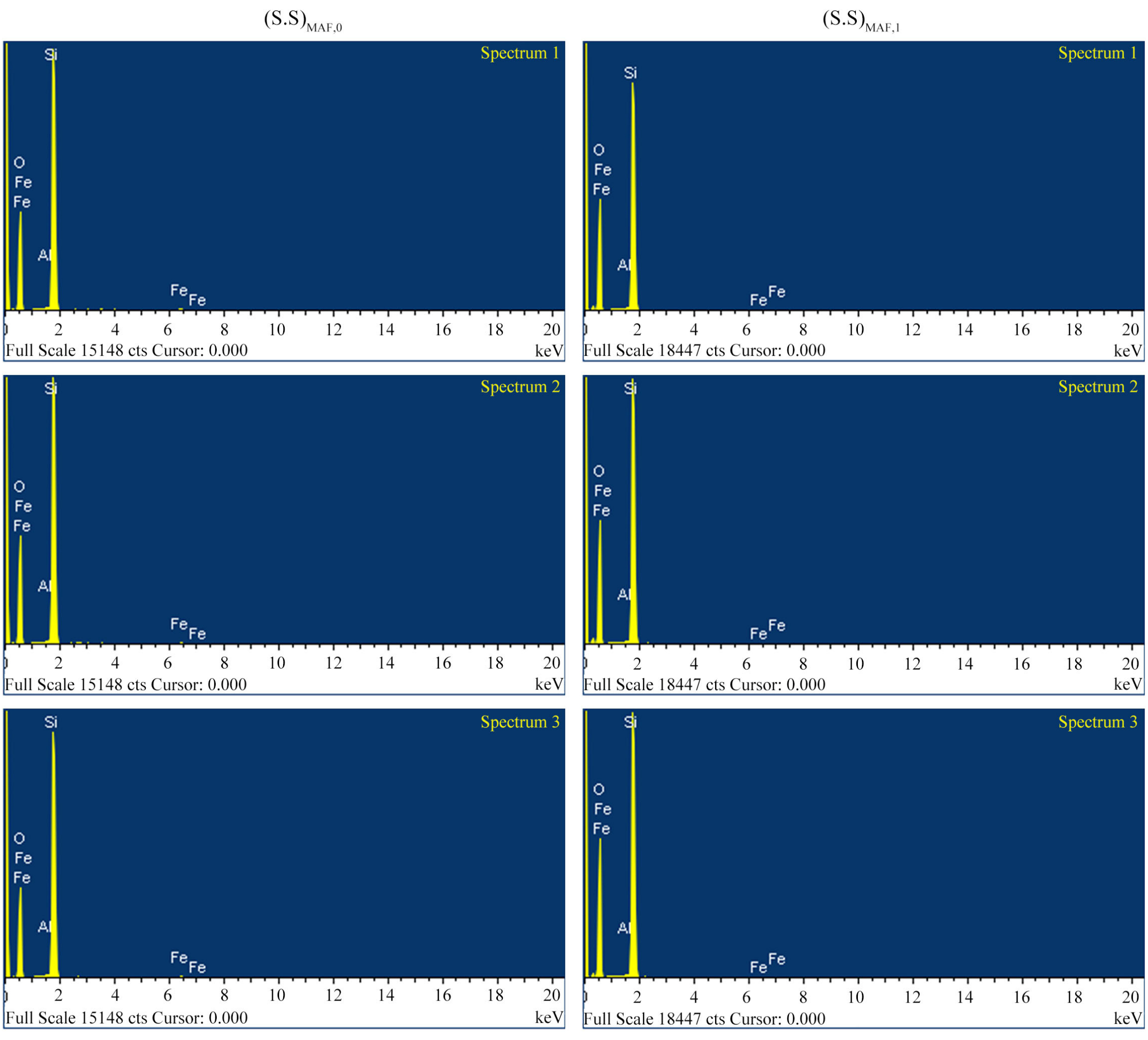


Figure 8. Energy dispersive spectra (EDS) of Maféré sand samples.

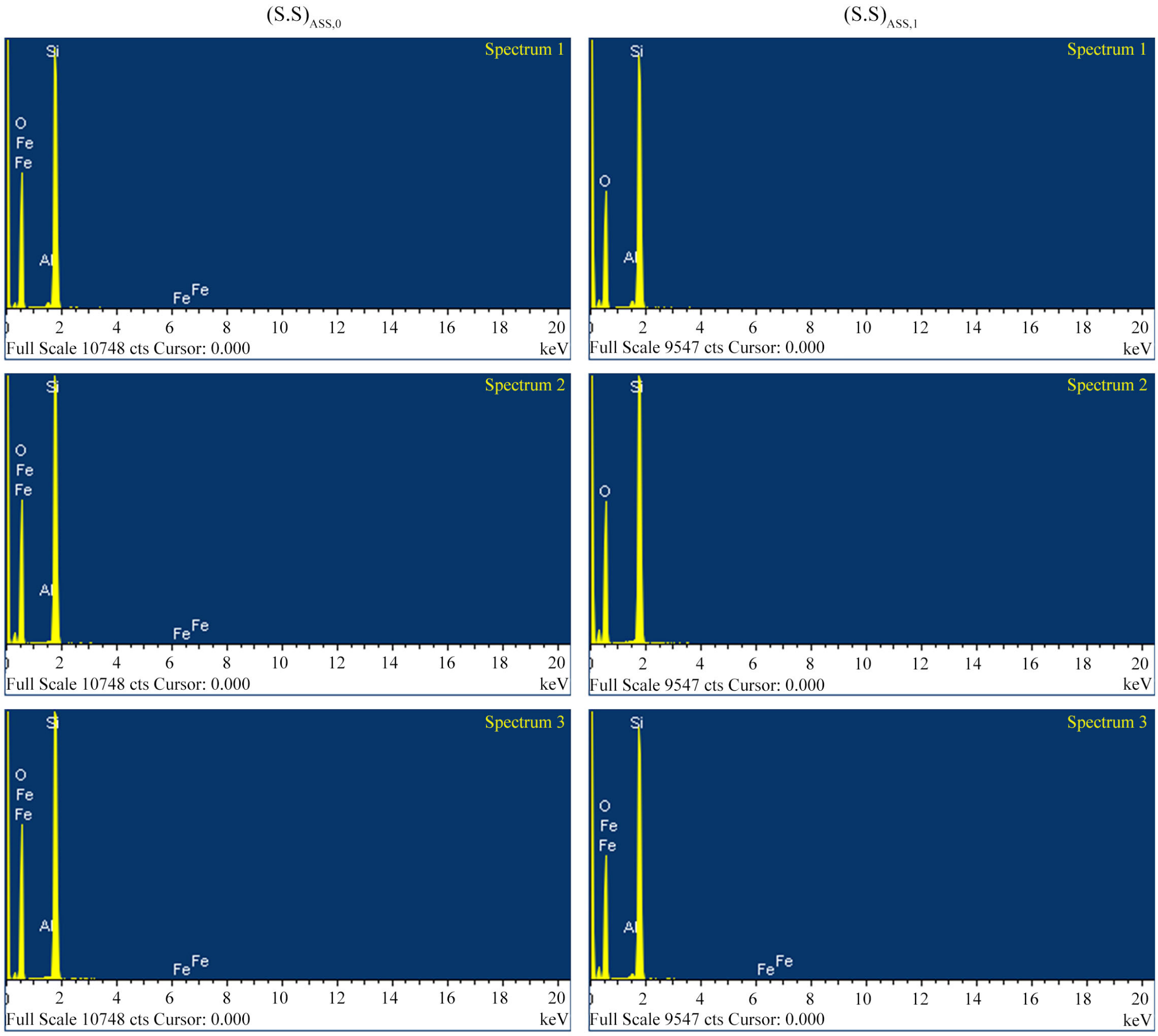


Figure 9. Energy dispersive spectra (EDS) of sand samples from Assinie.

The energy bands (in yellow) are characteristic of the chemical compounds present in the samples. Iron (Fe) appears at several locations in these spectra. The multiple appearance of iron is explained by the fact that its detection depends on its degree of oxidation. Thus, it can be FeO, Fe_2O_3 and Fe_3O_4. However, the proportion of iron in the samples is the accumulation of the rates of these different forms of oxidation detected.

Processing weight efficiency

The results of the performance evaluation of the treatment method employed using the formulas in Table 3 and Table 4 are in Table 10.

Wet sieving with attrition increased the silica content from 98.73% to 99.92%, a yield of 93.7% for the Maféré sand, while for the sand of Assinie, the proportion of silica increases from 98.82% to 99.44%, a yield of 52.53% (see Table 8). The rate of impurities in the sand of Maféré is reduced from 0.94% to 0.03%, a yield of 96.81% for iron oxide, and a reduction from 0.33% to 0.06%, a yield of 81.82% for alumina.

Table 10. Performance of the treatment method used.

Compound	Processing weight efficiency	
	(MAF): Maféré Sample	(ASS): Assinie Sample
Silica	93.70%	52.53%
Iron oxide	96.81%	76.92%
Alumina	81.82%	53.68%

In the sand of Assinie, the rate of impurities is also reduced after treatment. The content of iron oxide is reduced from 0.23% to 0.13%, a yield of 76.92%. A reduction from 0.95% to 0.44% is noted for alumina, a yield of 53.68%.

Considering these results, we note that the treatment technique used allows the increase in the content of silica while reducing that of the other oxides. Better results were obtained in this study with the sand of Maféré compared to the result obtained by Marouan Khalifa *et al.* (2019) in their process to produce high purity silica sand by heat treatment and acid leaching process [21]. In addition, all our treated samples have a higher silica content (at least 99%) than that of the samples obtained (98.1%) in the work of Sundararajan *et al.* (2009) [13].

In the following, we present images of the samples. Also, the grain morphology of the sands was evaluated using scanning electron microscope (SEM).

Micrography and Exoscopy of quartz grains

Figure 10 and Figure 11 are images of the raw and processed samples from Assinie and Maféré. These images are visual indicators of the purification of these sands.

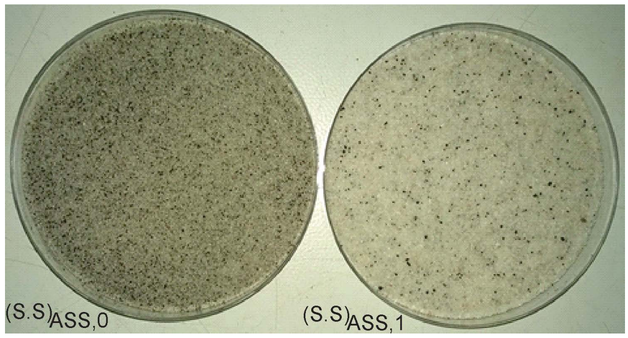
It was observed that the treated samples reflect more brightness (matte, vitreous) and color (white) of quartz (the natural silica). In the raw sand of Assinie, the treatment allows a notable reduction (as seen in Figure 10) of the organic matter (dark fraction)

The raw sand from Maféré has less organic matter than the sand from Assinie (Figure 11). However, there is more clay (very fine phase) in the sand of Maféré. The treatment reduces the proportion of clay and provides a sand containing clean quartz grains.

In addition, the SEM images below indicates the presence of different shapes of sand with irregular morphologies, for instance Figure 12 and Figure 13 show some rounded grains, whereas others are sub-rounded or elongated and sub-angular.

Generally, this indicates that the grains of these sands have undergone long transport, thereby, falls into the category of sands with blunt grains. Thus, these detritus have an origin further upstream in the crystalline basement of the Côte d'Ivoire.

Furthermore, the surface of the quartz grains observed at X500 magnification (see Figure 12(b); Figure 13(b)) is marked by numerous traces. The existence of these traces suggests a displacement of these grains in a turbulent environment. The primary deposits of these quartz grains are found in the basement rocks further upstream from the sampling sites.

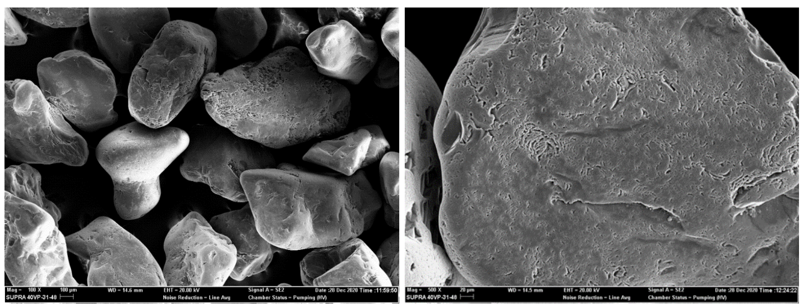


(a) (b)

Figure 10. Photograph of raw (a) and processed (b) sample from Assinie.

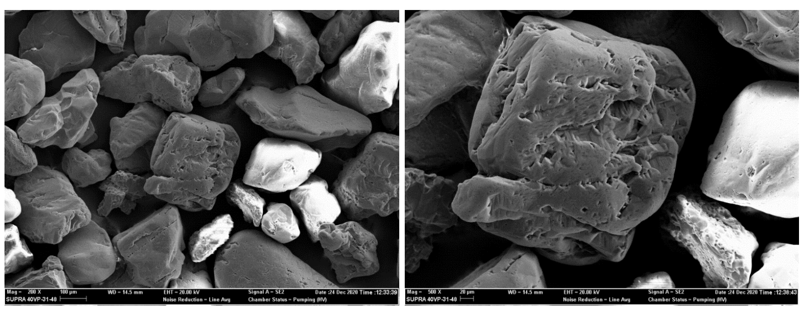
(a) (b)

Figure 11. Photograph of raw (a) and processed (b) sample from Maféré.



(a) (b)

Figure 12. Exoscopy of quartz grains from the Assinie sand. (a) 100X magnification; (b) 500X magnification



(a) (b)

Figure 13. Exoscopy of quartz grains from the Maféré sand. (a) 100X magnification; (b) 500X magnification.

4. Conclusions

We have carried out a purification treatment of sand samples taken from the sedimentary basin of Ivory Coast in West Africa. The objective of this study was to obtain silica sand ideal to produce glazing glass. Wet sieving and attrition technique were implemented in this purification process. The results from the analysis of the chemical composition of the raw and treated samples show a significant increase in the silica content and a significant reduction of impurities. The silica content (SiO_2) of the Maféré sand increased from 98.73% ± 0.15% to 99.92% ± 0.05%, *i.e.*, a yield of 93.70%, while of Assinie increased from 98.82% ± 0.67% in the raw samples to 99.44% ± 0.27% after treatment, a yield of 52.53%. The rate of iron oxide is reduced from 96.81% for the sand of Maféré against 76.92% for that of Assinie. Also, the proportion of alumina is reduced by 81.82% against 53.68% for the sand of Maféré and Assinie respectively. The sand of Maféré contains 53.2% of grains smaller than 500 microns and that of Assinie contains 29.30%. The technique of treatment used is more effective for the sand of Maféré containing more clay. Regarding the chemical composition of the said purified sands, they comply with the standard BS2975s of the American Ceramic Society and the National Bureau of Standards for the manufacture of window glass.

These results pave the way for feasibility studies for the opening of a sand quarry on these sites.

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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