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Research Article

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Physico-chemical characterization of marble from Assiyo (Dassa-Zoumè) in Benin

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Abstract The valorization of the natural resources located in Benin Republic, in particular, marble obtained from Assiyo (Dassa-Zoumè) was the purpose of this study. It was characterized to elucidate its properties for industrial applications. The results of the analysis by X-ray diffraction, infrared spectroscopy, X-ray fluorescence and thermal analysis showed that Assiyo marble is dolomitic limestone. This marble sample is basic with pH \approx 8.6. The particle size analysis of the powder particles of these marbles showed two major size fractions: 35 % of the particles have a size between 250 µm and 500 µm and 23 % of the particles have a size between 125 µm and 250 µm. These characteristics make this marble very good additives that can be used in the production of fertilizer, glass, cement, mortar, concrete and functional construction material. Assiyo marble can be used in metallurgy and as dolomite-based sorbent to remove pollutants from water.

Keywords marble; characterization; calcite; dolomite

1. Introduction

Marble is a naturally occurring stone mainly used in the construction industry [1][2][3][4][5] or as a raw material for artwork [6]. Since antiquity, it is one of the most frequently used materials for architecture and sculpture and therefore has been given an exceptional importance, particularly under the Romans [5][6]. The major phases constituting marble are either calcite (CaCO₃), or dolomite [CaMg(CO₃)₂] or both when they coexist in chemical equilibrium [2][3].

Some recent studies carried out by Topcu et al [7], Alyamac and Ince [8] showed that four different marble dusts produced in Turkey could be successfully and economically utilized as fillers in self-compacting concrete. Corinaldesi et al. [9], reported that marble powder proved to be very effective in assuring very good cohesiveness of mortar and concrete [9][10][11], even in the presence of a superplasticizing admixture [12], provided that water to cement ratio was adequately low.

Powdered marble can also be used as calcium and magnesium-based adsorbents for pollutants removal from drinking water, such as lead (II) ions [13][14][15][16][17], fluoride ions [18][19][20][21], copper (II) ions [22], cadmium (II) ions [16][17][23], zinc (II) ions [17], arsenic (III) [24], arsenate [25], strontium (II) [26], barium (II) [26], nickel (II) [23], catechol [27] and methylene blue [28][29].



According to OBRGM (Office Béninois des Recherches Géologiques et Minières) [30], the Assiyo marble deposit is currently being assessed in order to know the quantity. From available literature, studies have mainly been concentrated on the thermophysical properties of granite, marble and basalt deposits in Benin. They reported that granite is more insulating whiles marble has better ability to store heat [31]; however, no physicochemical characterization of the marbles were undertaken. The physical and chemical properties of natural stones play an important role in choosing their field of application especially as building stone.

This study carefully presents the results of the physicochemical characterization of eight marble samples taken from different regions of the Republic of Benin in order to demonstrate their different potential applications. X-ray diffraction analysis, granulometric study, thermal analysis, chemical analysis by X-ray fluorescence and infrared spectroscopy have been used to characterize this sample for its physical, thermal, chemical and microstructural properties.

2. Materials and Methods

2.1 The Study Area and Sample Collection

The marble sample was collected from deposit site of Assiyo (Dassa-Zoumè) in Benin. Information on the sample collected and the sampling areas are presented in Table 1 and Fig 1 respectively.

Table 1: The names of the village, municipality, department, and code of the sample studied

Department	Municipality	Village	Sample code	
COLLINES	Dassa-Zoumè	Assiyo	ASS	

After the sample collection in marble deposit site, the primary marble blocks were coarsely crushed into secondary marble blocks and taken to the laboratory for further evaluations.



Figure 1: Map of Benin showing sample location



These secondary marble blocks were further crushed into coarse marble particles (aggregates) using a mass hammer mill to facilitate crushing: this is known as secondary crushing. These obtained marble particles were plundered in an aluminum mortar and then finely ground in a ceramic mortar. The marble powder obtained was sifted through a 500 micrometers mesh sieve. The sifted marble powder (*see Fig 2*) was stored at room temperature in clean plastic bottles.



Figure 2: Pictures of marble and powder obtained after crushing

2.2 Methods

For the granulometric analysis, about 200 g of marble powder sample was sieved and separated into six different fractions by using Haver & Boecker sieves with mesh sizes ranging from 500 μ m to 50 μ m. With the aid of a Mettler-Toledo pH-meter, the pH of sample was determined according to the method used by Emofurieta et al [32]. Typically, 5 g each of crude marble powder was placed in two s 50 mL bottle of distilled water and magnetically stirred for 24 h and 48 h respectively. After this time, the colloidal solution was left standing for 12 hours to allow the settling of the marble suspensions; after which the pH measurement was performed on water supernatants [33]. Five separate tests were carried out and the average values taken.

The percentage mass of calcium carbonate in the powdered marble sample were measured using the back titration methodology.1 g of each powdered marble sample was weighed and placed in labelled 250 mL conical flasks. Few drops of ethanol were added to the flasks to act as a wetting agent and catalyst for acid reactions. 50 mL of HCl solution (1.0 M) was then added to each of the labeled conical flasks, and swirled well to completely wet all the solids. The solutions in the flasks were magnetically stirred for 5 hours to allow the total dissolution of the carbonate (CaCO₃) contained in the powder according to the reaction showed in equation (1).

$$CaCO_3 + 2(H_3O^+ + Cl^-) \rightarrow (Ca^{2+} + 2Cl^-) + CO_2 + 3H_2O.$$
 (1)

3-4 drops of phenolphthalein indicator were added to the flasks, whiles excess of unreacted acid was titrated against standardized NaOH solution (2 M), until barely pink color appears, persists for 30 s and fades slowly. The titration experiments were repeated 5 times to obtain concordant values. The mass percent of calcium carbonate in the powdered marble samples was determined using equation (2)

$$\%(\text{CaCO}_3) = \frac{\text{m}(\textit{CaCO}_3)}{\text{m}(\text{marble powder})} \chi \ 100$$
(2)

X-ray powder diffraction (p-XRD) measurements were performed using a Philips diffractometer (PANalytical, X'pert Pro MPD model) with a Bragg-Brentano configuration equipped with a Cu-K α radiation (1.54059 Å) tube operated at a voltage of 45 kV and current of 40 mA. The data of the randomly arranged powder sample were



collected in the 5 °- 80 ° 2 θ value range with scan speed of 0.042°/sec, step size of 0.01° and a count time of 10.0 s per step. The diffraction patterns were matched against the ICDD's PDF database and qualitative phase analysis conducted using the X'Pert Highscore plus search match software (Panalytical, Netherlands).

The chemical composition of the marble sample was determined using a THERMOFISHER ARL PERFORM'X 2.5KW X-ray fluorescence spectrometer. The sample studied was analyzed in the form of pellets. Thus, 2 g of very fine powder are shaped into a uniform pellet with a diameter of 25 mm using a mold under a pressure of 8 to 15 tons.

Chemical bonding was analyzed by transmission infrared spectroscopy, using a Perkin Elmer 100 Series Fourier Transform Infrared (FTIR) spectrometer. Powdered marble sample was finely ground in a mortar and then mixed with potassium bromide (KBr) powder (1/100 by weight). The powder mixture was put in a 13 mm diameter mould and pressed at high pressure using a hydraulic press to form thin pellets. In order to minimize the amount of water adsorbed, the pellets were heated in a furnace overnight at 100 °C. Spectra data were recorded and analysed using the Spectrum 10TM software within the 4000 - 450 cm⁻¹ spectral range in transmission mode.

Thermal analysis, i.e. thermogravimetry (TG) and differential thermal analysis (DTA), were performed by using a SETARAM LABSYS system, from ambient temperature to 1000 °C, using a 20 °C/min heating rate and a 100 ml/min nitrogen flow. The measurement was made using about 8 mg of powdered marble in a 100 μ L alumina crucible.

3. Results and Discussion

3.1 Measurements of pH and carbonate content

The pH of the suspensions indicates the level of alkaline or acidic species contained in the marble powder. The average pH values of the ASS marble sample (Fig.3) are approximately equal to 8.6 for suspensions stirred for 24 h and 48 h. These measured pH values are therefore higher than the neutral pH value (7.00), showing that suspensions are basic: an indicator of the presence of alkaline species in the ASS marble powder. These results are close to those reported by Ahmed et al [11], on the potential use of marble and granite solid wastes as environmentally friendly coarse particulate in civil construction.





The percentage carbonate content in the ASS marble sample is presented in Fig 4. This result revealed high carbonate content (72.4 wt.%) for ASS sample. This suggests that ASS sample is carbonated.





Figure 4: Carbonate content of the ASS marble sample

3.2 Particle Size Distribution

From the particle size distribution results presented in Fig. 5, the ASS sample could be categorized into three size fractions. The first size fraction is in the range of 250 μ m and 500 μ m, the second size fraction is between 125 μ m and 250 μ m; and the third size fraction is between 63 μ m and 125 μ m. These results showed that for the marble powder sample, only about 19 wt.% of the grains have a size < 63 μ m and therefore, can be used to reinforce concrete.



Figure 5: Histogram of the granulometric repartition of the ASS marble powder

3.3 X-ray Diffraction (p-XRD)

Fig. 6 shows the p-XRD patterns of the ASS marble sample. The diffraction peaks of ASS sample were matched with ICDD PDF-no. 01-085-1108 and 98-008-7088 which indicated the presence of Calcite (CaCO₃) and Dolomite



 $[CaMg(CO_3)_2]$, respectively. The main reflections of calcite and dolomite have almost the same intensity for ASS marble. Attributions of calcite and dolomite reflections are in good agreement with those reported on marbles characterization [2][3][9][34][35][36][37].



Figure 6: XRD pattern of ASS marble sample

3.4 XRF chemical analysis

The chemical composition of the IDA marble sample was determined by an X-ray fluorescence spectrometer. The analysis revealed the results grouped in Table 2 below.

Table 2. Chemical composition of the studied marble powder sample (% by weight).					weight).				
Sample	Composition in %								
	CaO	MgO	SiO ₂	Al_2O_3	Fe_2O_3	K ₂ O	Na ₂ O	LOI	TOTAL
ASS	30.65	19.18	13.86	00.36	00.55	00.01	00.07	34.93	99.61

According to Table 2, high amounts 30.65% and 19.18% of calcium (CaO) and magnesium (MgO) oxides respectively were observed, showing that ASS essentially contain dolomite or calcite (CaCO₃) and dolomite [CaMg(CO₃)₂] [24][27][38]. The loss on ignition, symbolized by L.O.I., essentially represents carbon dioxide (CO₂) from the calcite and dolomite contained in this sample and revealed by X-ray diffraction. The percentage of other oxides (SiO₂, Al₂O₃, K₂O, Na₂O and Fe₂O₃) is negligible (almost zero) except for the silica (SiO₂) contents which reached 13.86%.

3.5 Fourier Transformed Infrared Spectroscopy

Fig. 7 shows the FT-IR analysis spectra of the ASS marble sample. The main bands of absorption obtained from Fig. 6, are regrouped in Table 3.

Table 3 Main bands of FT-IR absorption and associated bond vibration of ASS marble sample

Fundamental frequencies (cm ⁻¹)		Modes of vibration		
713	ν_4	In-plane bending mode of CO ₃ ²⁻		
877	v_2	Out of plane bending mode of CO ₃ ²⁻		
1414	v ₃	Asymmetric stretching mode of CO ₃ ²⁻		
1810	$v_1 + v_4$	Combination of vibrations of v_1 and v_4		
3470	ν	Symmetric OH stretch in H-O-H		

The spectra of ASS marble sample shows the bands observed at 713 cm⁻¹, 877 cm⁻¹, the broad band at 1414 cm⁻¹ and 1810 cm⁻¹ representing vibration bands of carbonate radicals which is due to the presence of dolomite and calcite mineral [39][40] in the ASS marble sample. This result corroborated with those obtained by carbonate content



(section 3.1), XRD (section 3.3) and XRF (section 3.4) in this study, revealed the presence of mixture of calcitedolomite in the ASS marble.



Figure 7: FT-IR spectra of ASS sample

3.6 Thermogravimetric/Differential Thermal Analyses

The TGA/DTA curves show the thermal decomposition under nitrogen flow for the ASS sample.

The curve of the ASS sample (in Fig. 8) shows a loss of mass. This mass loss begins slowly at 350 °C. It goes from 350° C up to 650° C (loss of 4.2 %) and increases between 650° C and 825° C with a loss of 29.1 %, i.e. a total of 33.3 %. The mass loss observed at the level of ASS is low compared to the others because it contains silicon oxide (SiO₂) up to 13.86 %. As for the DTA curves, an endothermic peak is observed at 800 °C with a splitting at 710 °C which shows in addition to the presence of calcite, the presence of dolomite which decomposes in two stages [27][41][42][43][44][45][46][47][48]. The resolution at 710 °C indicates the decomposition of the magnesium carbonate contained in the dolomite into magnesium oxide (MgO) and carbon dioxide (CO₂). The endothermic peak at 800 °C indicates the decomposition of calcium carbonate from calcite and dolomite. These different reactions give the following equations:

$$\begin{array}{rcl} CaCO_3 & \rightarrow & CaO & + & CO_2 \\ CaMg(CO_3)_2 & \rightarrow & CaO + MgO + 2CO_2 \end{array} \tag{3}$$





Figure 8: TG-DTA curves of ASS marble sample

4. Conclusion

The following conclusions have been drawn from this study: ASS marble is dolomitic limestone. This sample is basic (pH \approx 8.6). The particle size study of the powder of this sample shows two major size fractions: 23 % of the particles have a size between 125 µm and 250 µm and 35 % of the particles have a size between 250 µm and 500 µm.

The characteristics obtained make this marble very good additives that can be used in the production of fertilizer, glass, cement, mortar, concrete and functional construction material. ASS marble can be used in metallurgy and as dolomite-based sorbent to remove pollutants from water.

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