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Adsorption Isotherm of BET Nitrogen of Concretes with Consolidated Soil by Sugar Cane Molasses

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Abstract: Sugar cane molasses is often poured out on roads with soil in the city of Nkayi, Republic of Congo in order to reduce the dust. Nitrogen physical adsorption has allowed us to collect information on the state of the accessible total area according to the quantity of sugar cane molasses. The adsorption isotherms, the specific area, the adsorbed quantity of nitrogen on a Q_m mono layer, the number of molecules constituting the adsorbed sugar cane molasses (n') have been examined. The obtained results show that the quantity of sugar cane molasses in the material does not modify the adsorption isotherm of nitrogen of type IV that remains and a hysteria loop of type H4 in all samples, this justifies the monocoat-multicoat adsorption mechanism with capillary condensation and mesopores presence in the structure of materials. Materials with elaborated raw soil by clayey fine soil used are mesoporous materials. More of 50×10^{18} molecules constituting sugar cane molasses occupy the extreme area accessible to soil clay, without occupying on accessible sites.

Key words: Isotherm, adsorption, BET (Brunauer, Emmet, and Teller) specific area, clayey fine soil, sugar cane molasses.

1. Introduction

Sugar cane molasses is used during the dry season to reduce the dust on roads by soil in the city of Nkayi, Republic of Congo (see in Fig. 1).

It has proved a cohesive excellent performance on aggregates (fine particles) of the soil of the city of Nkayi. Roads by treated soil with sugar cane molasses solution remain stable (no dust spreading) during at least 4 weeks while roads by soil dried with simple water remain are stable (no dust spreading) in a few time, about an hour or half hour time (report done on the ground).

The aptitude of sugar cane molasses consisting of bringing a seed of cohesion to the soil of the city of Nkayi, really finds its explanation at the ladder of the soil structure and at the chemical composition of sugar cane molasses.

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Malanda et al. [1] have observed an increase of resistance of bricks by consolidated soil with sugar cane molasses that brings a seed of cohesion.

Ngouallat et al. [2] have observed a change of dye on the consolidated soils by sugar cane molasses and have suggested a possible reaction between organic constituents of sugar cane molasses and active constituents of soil. That is, the clay [2]. Now, the specific area of clays is the place of possible interactions in clayey materials [3-5], thus, between clay and sugar cane molasses.

Nitrogen physical adsorption is a method that can allow collecting information on the state of the total accessible area and the porosity distribution in soil according to the quantity of sugar cane molasses.

Experienced results of nitrogen physical adsorption are given in the form of curves linking quantity of adsorbed nitrogen according to the relationship between partial pressure and saturating vapor pressure for nitrogen. These curves are called isotherms [6].

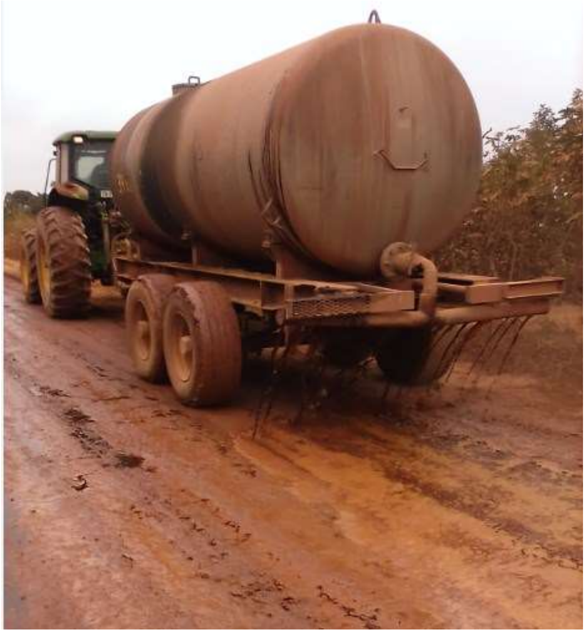

Fig. 1 Sugar cane molasses poured out on the road by soil in the city of Nkayi (Picture taken on the ground).

BET (Brunauer, Emmet, and Teller) pattern is employed for the determination of specific area, and the quantity of adsorbed gas on a monolayer [7, 8]. The used appliance is the Micromeritics ASAP 2460 Version 2.01.

2. Materials and Method

2.1 Materials

Sugar cane molasses and the clayey fine soil from both of the city of Nkayi, Republic of Congo are basic materials of our study.

2.1.1 Geotechnical Characterization on Taken Soil in Nkayi

The results of some realized geotechnical tests on taken soil in Nkayi are presented in Table 1.

According to the triangular abaque of Taylor, the soil of Nkayi is a clayey soil (Fig. 2).

Table 1 Geotechnical characterization on taken soil in Nkayi.

Materials	Granulometrical distribution				Atterberg limits			Compactibility		Blue of methylene
	% fines (< 80 µm)	clay < 2 µm	Silt between 2 µm and 63 µm	Soil between 63 µm and 2 mm	W_L (%)	W_p (%)	I_p (%)	γ_d (g/cm^3)	W (%) (OPM)	VBS (g/100 g)
Soil taken at 1 m deep	88	54	34	12	42	21	21	1,68	15	0.34

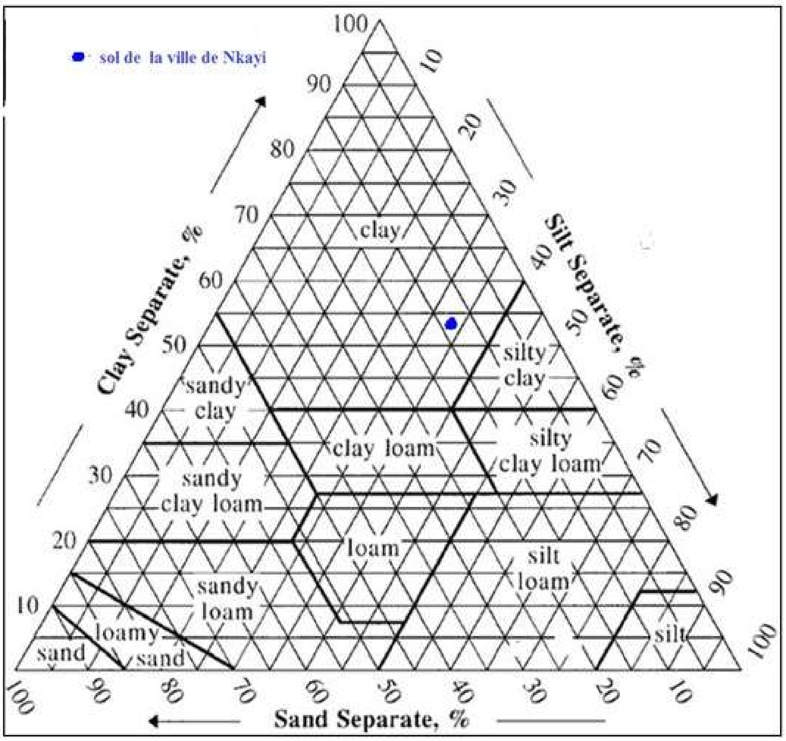

Fig. 2 Triangular abaque of Taylor, the soil of Nkayi.

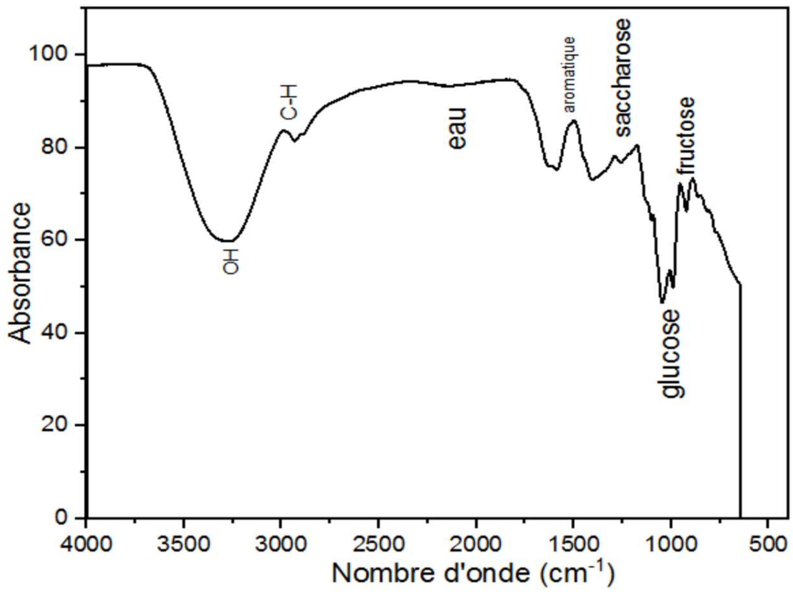

Fig. 3 Infraredspectrum of sugar cane molasses.

To sum up, the soil of the city of Nkayi is a fine clayey.

2.1.2 Sugar Cane Molasses

Chemical analysis by infrared of sugar cane molasses is given in Fig. 3.

The region situated between 1,200 and 1,000 cm^{-1} is characterized of glycosidic connections (C-O-C) in combination with other methods such as: (C-O-H), (C-C), (C-H) of polysaccharids, that is saccharose. The region between 950 and 1200 cm^{-1} is characterized of glucoses. The region between 950 and 400 cm^{-1} is characterized of fructoses. The pick at 1,582 cm^{-1} is characterized by the vibration of connections C=C, aromatic compound responsible, and of the sugar cane molasses coloration as the maltol and the furaneol. Our sugar cane molasses is composed of saccharase, glucose, fructose, water and of coloring compound such as themaltoland the furaneol. Phuong T. (2013). [9], Mathlouthi Mohamed(1998) [10] and Janekarn et al. [11] have done the same report.

2.2 Method

The pattern of BET (Brunauer, Emmett and Teller), known under the initials BET, and dated from 1938, is based on physical adsorption of nitrogen gas (N_2) at the low temperature (77 K) [9-11]. The analytic treatment from the experience of the determinate adsorption isotherm allows defining the quantity of absorbed gas in a full monolayer, and then calculating the area of that layer which is the specific area of the solid.

The equation of BET pattern is given by the following relationship [12, 13]:

$$Q = \frac{Q_m C(\frac{P}{P_0})}{\left(1-\frac{P}{P_0}\right)[1+(C-1)(\frac{P}{P_0})]} \quad (1)$$

where:

Q: The adsorbed quantity by adsorbing gram at the relative pressure $(\frac{P}{P_0})$;

Q_m: The required adsorbed quantity to form a monolayer of soluty;

C: The constant of BET.

The linear transformation of BET equation is given under the following form:

$$\frac{1}{Q\left(1-\frac{P}{P_0}\right)} = \frac{1}{Q_m C} + \frac{C-1}{Q_m C}(\frac{P}{P_0}) \quad (2)$$

By drawing experimental data under the form of

$\frac{1}{Q\left(1-\frac{P}{P_0}\right)}$ according to $(\frac{P}{P_0})$, the slope $(\frac{C-1}{Q_m C})$ and tidy at origin $(\frac{1}{Q_m C})$ of the linear regression of experimental points allow then calculating Q_m and the constant C [6].

The specific area of the sample is given by the following equation [3, 7]:

$$S_{(\mathrm{BET})} = \frac{Q_m \times N \times S_m}{22414} \quad (3)$$

with,

N: The number of Avogadro (6.023×10^{23} atoms/mol);

S_m: The occupied surface by a gas molecule at 77 K (m^2);

But, the specific area of the sample is given by the following equation [3]:

$$S_{(\mathrm{BET})} = \frac{n \times S_m}{m} \quad (4)$$

where:

n: Number of adsorbed gas molecules in monolayer;

m: The masse of sample (g).

The number molecules constituting the adsorbed sugar cane molasses in monolayer is calculated through the formula:

$$n = \frac{Q_m \times N \times m}{22414} \quad (5)$$

The number molecules constituting the adsorbed sugar cane molasses in monolayer is calculated through the formula:

$$n' = n_1 - n_i \quad (6)$$

with,

n_1: Number of molecules of adsorbed nitrogen gas in monolayer on the sample without sugar cane molasses (0%);

n_i: Number of molecules of adsorbed nitrogen gas in monolayer on the consolidated sample at i (%) of sugar cane molasses, with i = 8%, 12%, 16%.

3. Results and Discussion

3.1 BET Adsorption Isotherm of Nitrogen on Soil without Sugar Cane Molasses (0%)

Obtained experienced results are presented in Table 2.

The isotherm obtained (Fig. 4) using the BET model equation and the calculated parameters are presented in Table 6.

Table 2 Experimental results according to BET pattern.

Relative pressure (P/P_0)	Quantity adsorbed (cm^3/g STP)	$1/[Q\ (P_0/P-1)]$
0.051941454	7.1902	0.007620
0.084072676	7.7418	0.011856
0.119961406	8.2491	0.016525
0.154961545	8.7027	0.021072
0.189994871	9.1439	0.025652
0.224958685	9.5915	0.030262
0.259934856	10.0480	0.034955
0.294983578	10.5224	0.039763

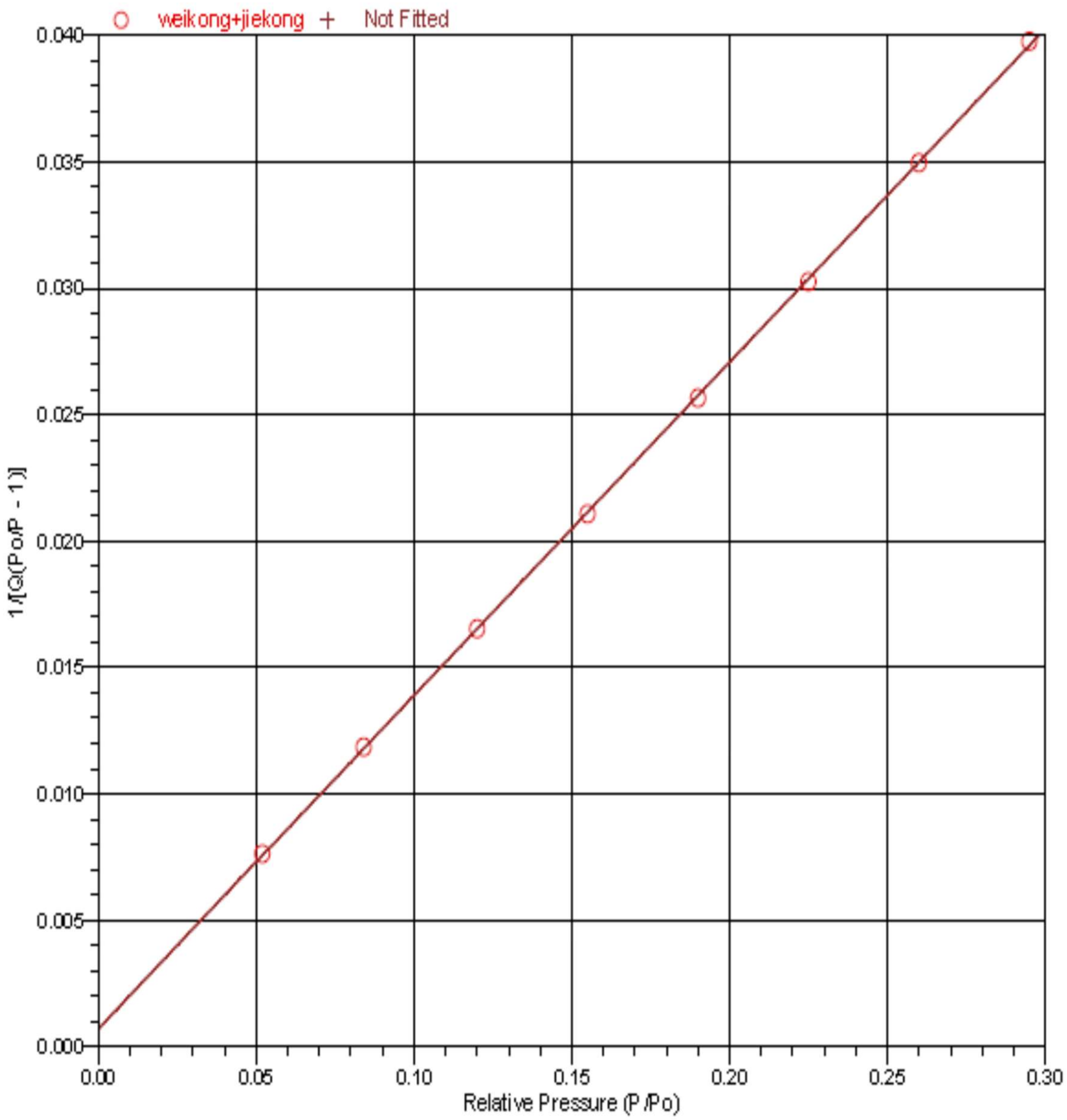

Fig. 4 BET pattern of the sample without sugar cane molasses (0%).

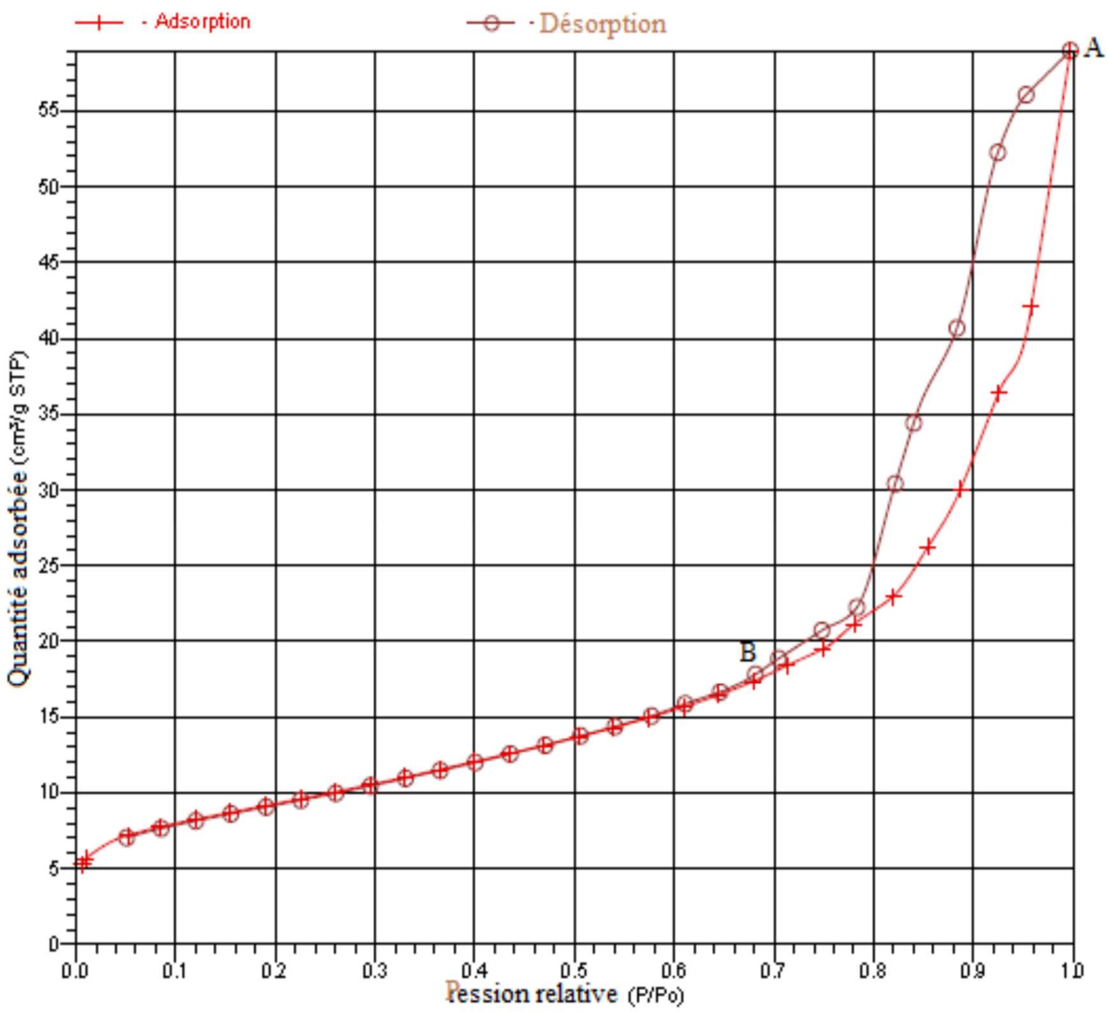

Fig. 5 Adsorption isotherm and nitrogen desorption on the sample without sugar cane molasses according to the relative pressure.

$P/P_0 < 0.1$ corresponds with filling of micropores. $0.1 < P/P_0 < 0.4$: formation of monolayer on the partitions of mesopores. $0.4 < P/P_0 < 0.6$: transition mono and multilayer. $0.6 < P/P_0 < 0.8$: multilayer adsorption on partitions of mesopores. $0.8 < P/P_0 < 0.9$: capillary condensation on the white of adsorption (the gas is condensed in liquid mesopores jumbled together with formation of a gas-liquid meniscus) and the evaporation on the white of desorption. The formation of the hystersis loop occurs between $0.68 < P/P_0 < 0.99$.

The Figs. 5 and 6 show that the adsorption isotherm and the nitrogen desorption on the sample without sugar cane molasses (0%) correspond to isotherm of type IV that suggests the presence of diameter pores between 20 and 500 Å (mesopores) [16]. The hysteresis existence during the desorption reflects the presence of mesopores in which the vapor is condensed by forming a strong curvature of meniscus [17].

The opening point (A) and closing point (B) of the hysteresis loop are:

- On the adsorption-desorption isotherm as a function of relative pressure (Fig. 5)

A (0.99, 59 cm^3/g); B (0.69, 17 cm^3/g)

- on the adsorption-desorption isotherm as a function of absolute pressure (Fig. 6)

A (760 mmHg, 59 cm^3/g); B (500 mmHg, 17 cm^3/g)

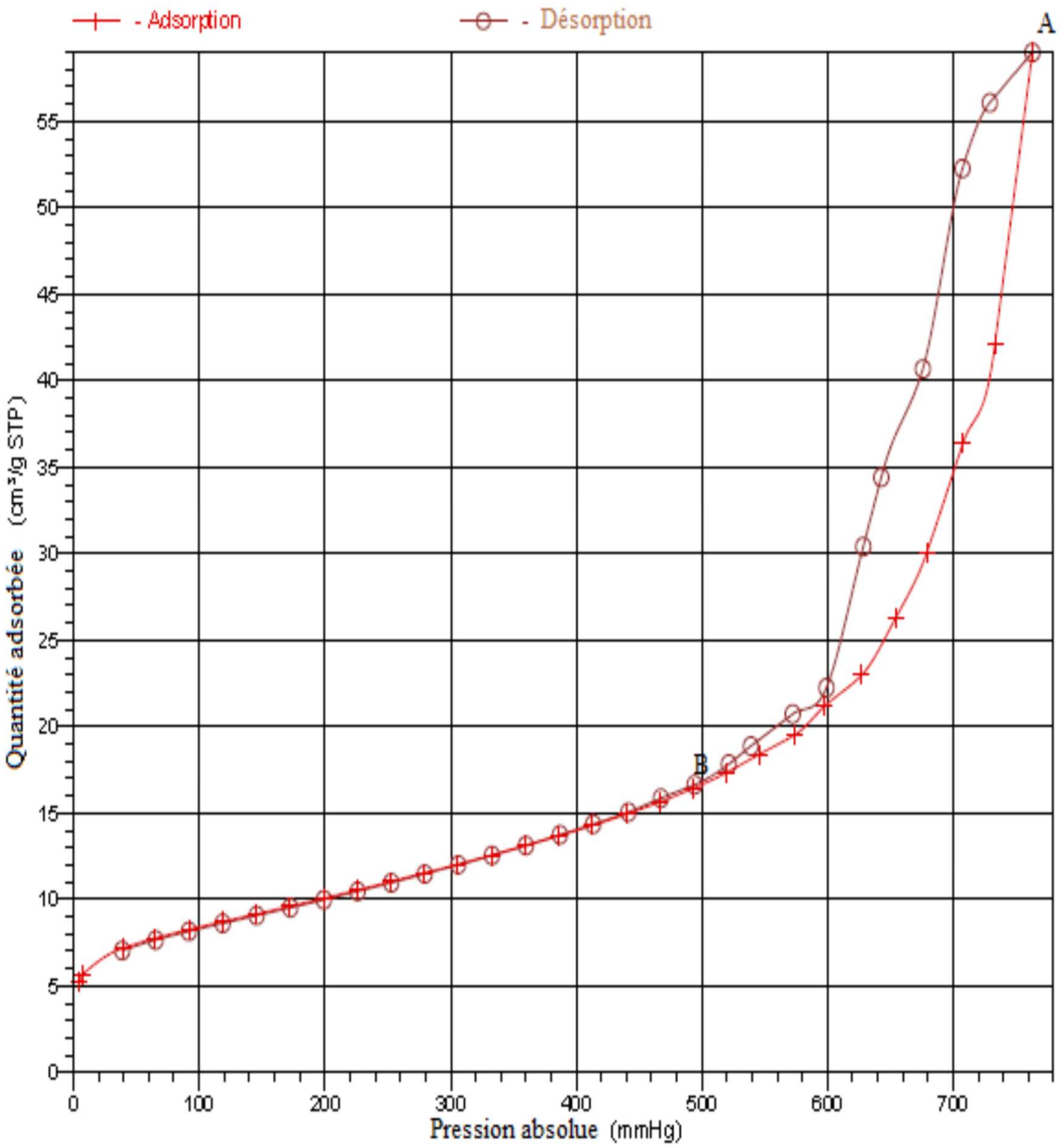

Fig. 6 Adsorption isotherm and nitrogen desorption on the sample without sugar cane molasses (According to the absoluted pressure.

3.2 BET Adsorption Isotherm of Nitrogen on Consolidated Soil at 8% of Sugar Cane Molasses

The obtained experienced results are presented in Table 3.

The isotherm obtained (Fig. 7) using the BET model equation and the calculated parameters are presented in Table 6.

Table 3 Experienced results for the application of BET pattern.

Relative pressure (P/P_0)	Quantity adsorbed (cm^3/g STP)	$1/[Q(P_0/P-1)]$
0.053200087	4.5934	0.012233
0.084289689	5.0314	0.018295
0.120086113	5.3979	0.025283
0.155086878	5.6996	0.032204

Table 3 to be continued

0.190153596	5.9663	0.039355
0.225230501	6.2252	0.046699
0.260217076	6.4735	0.054337
0.295188900	6.7131	0.062388

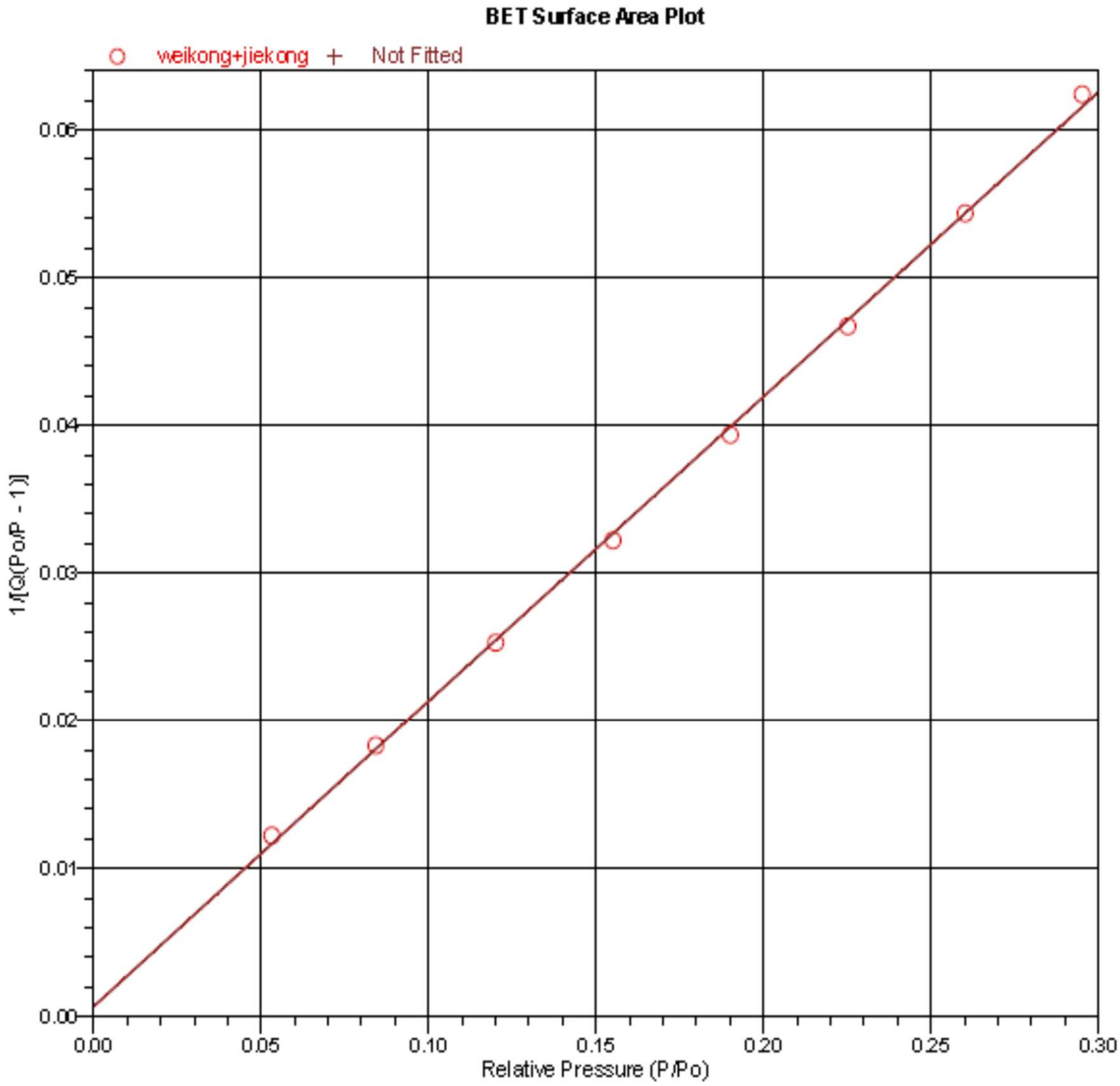

Fig. 7 BET pattern of the consolidated sample at 8% of sugar cane molasses.

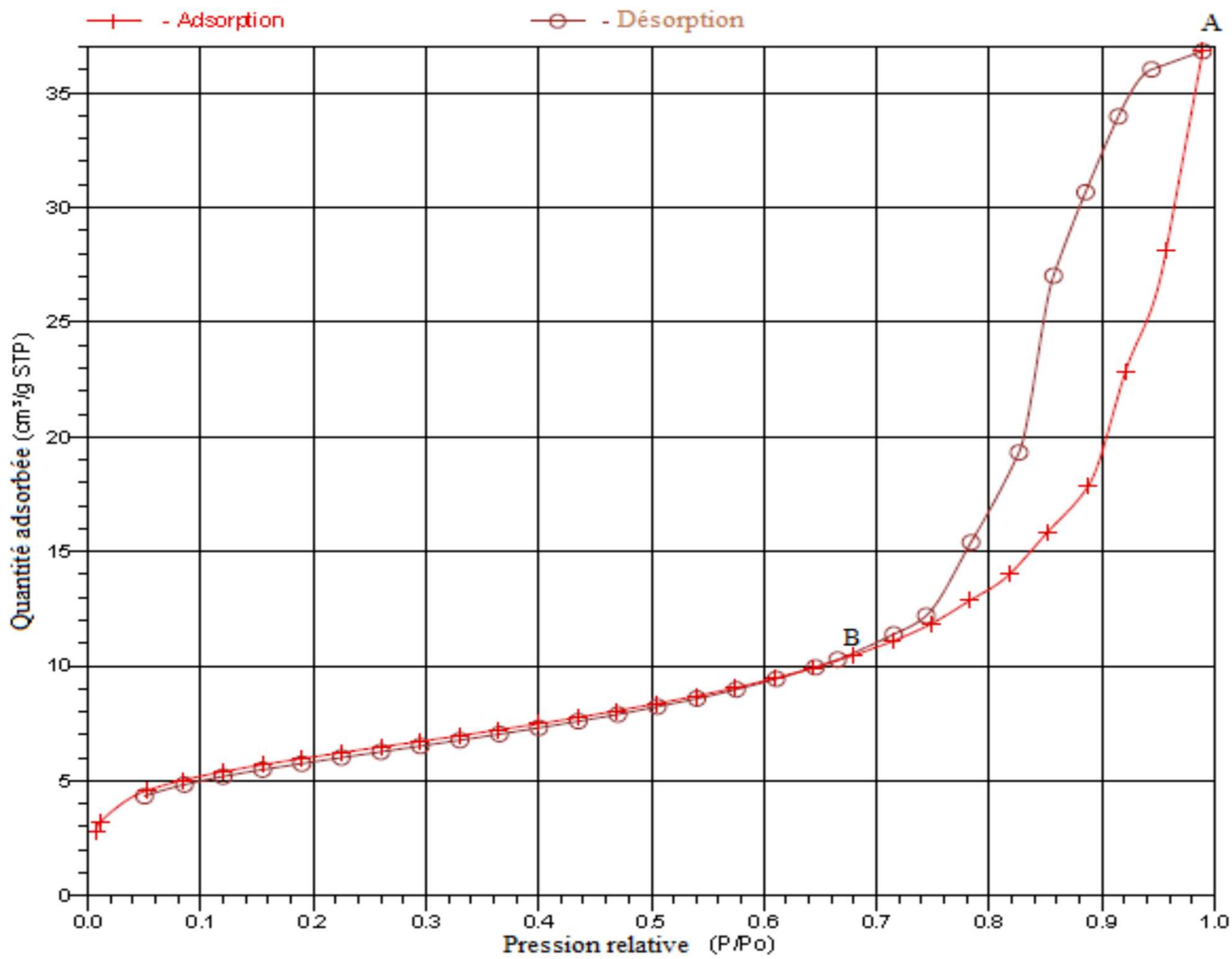

Fig. 8 Adsorption isotherm and nitrogen desorption on the consolidated sample at 8% of sugar cane molasses. According to the relative pressure.

The Figs. 8 and 9 show that adsorption isotherm and nitrogen desorption on consolidated sample at 8% of sugar cane molasses correspond to isotherm of type IV that suggests the presence of pores (mesopores). The opening point (A) and closing point (B) of the hysteresis loop are:

- On the adsorption-desorption isotherm as a function of relative pressure (Fig. 8)

A (0.99, 37 cm^3/g); B (0.68, 11 cm^3/g).

- on the adsorption-desorption isotherm as a function of absolute pressure (Fig. 9). A (760 mmHg, 37 cm^3/g); B (520 mmHg, 10.5 cm^3/g).

The opening point (A) of the hysteresis loop defines the maximum adsorbed amounts and the maximum pressure. The 8% molasses stabilized sample adsorbs less gas (37 cm^3/g) than the 0% sample (59 cm^3/g), although both samples reached the same maximum pressure (760 mmHg). This suggests that the 0% sample is more porous than the 8% sample.

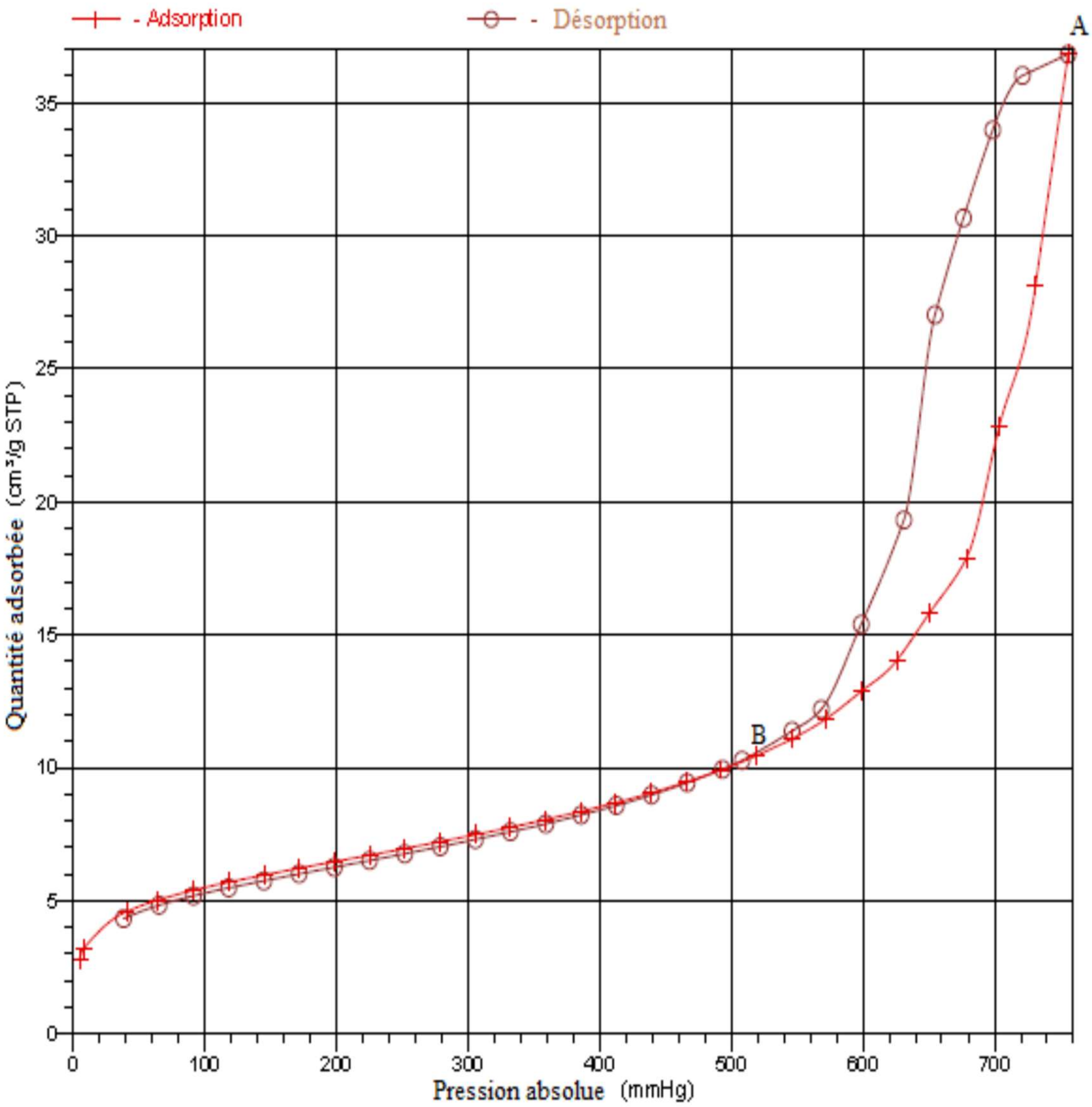

Fig. 9 Adsorption isotherm and nitrogen desorption on the consolidated sample at 8% of sugar cane molasses. According to the absolue pressure.

3.3 BET Adsorption Isotherm of Nitrogen on the Consolidated Soil at 12% of Sugar Cane Molasses

The obtained experienced results are presented in Table 4.

Table 4 Experienced results for the application of BET pattern.

Relative pressure (P/P_0)	Quantity adsorbed (cm^3/g STP)	$1/[Q(P_0/P\text{-}1)]$
0.053088130	3.8565	0.014538
0.084205408	4.2626	0.021571
0.120032654	4.6149	0.029558
0.155073316	4.9030	0.037433
0.190125079	5.1645	0.045456
0.225129597	5.4134	0.053671
0.260246650	5.6529	0.062234
0.295153841	5.8992	0.070984

The obtained isotherm (Fig. 10) from BET pattern equation and calculated parameters are presented in Table 6.

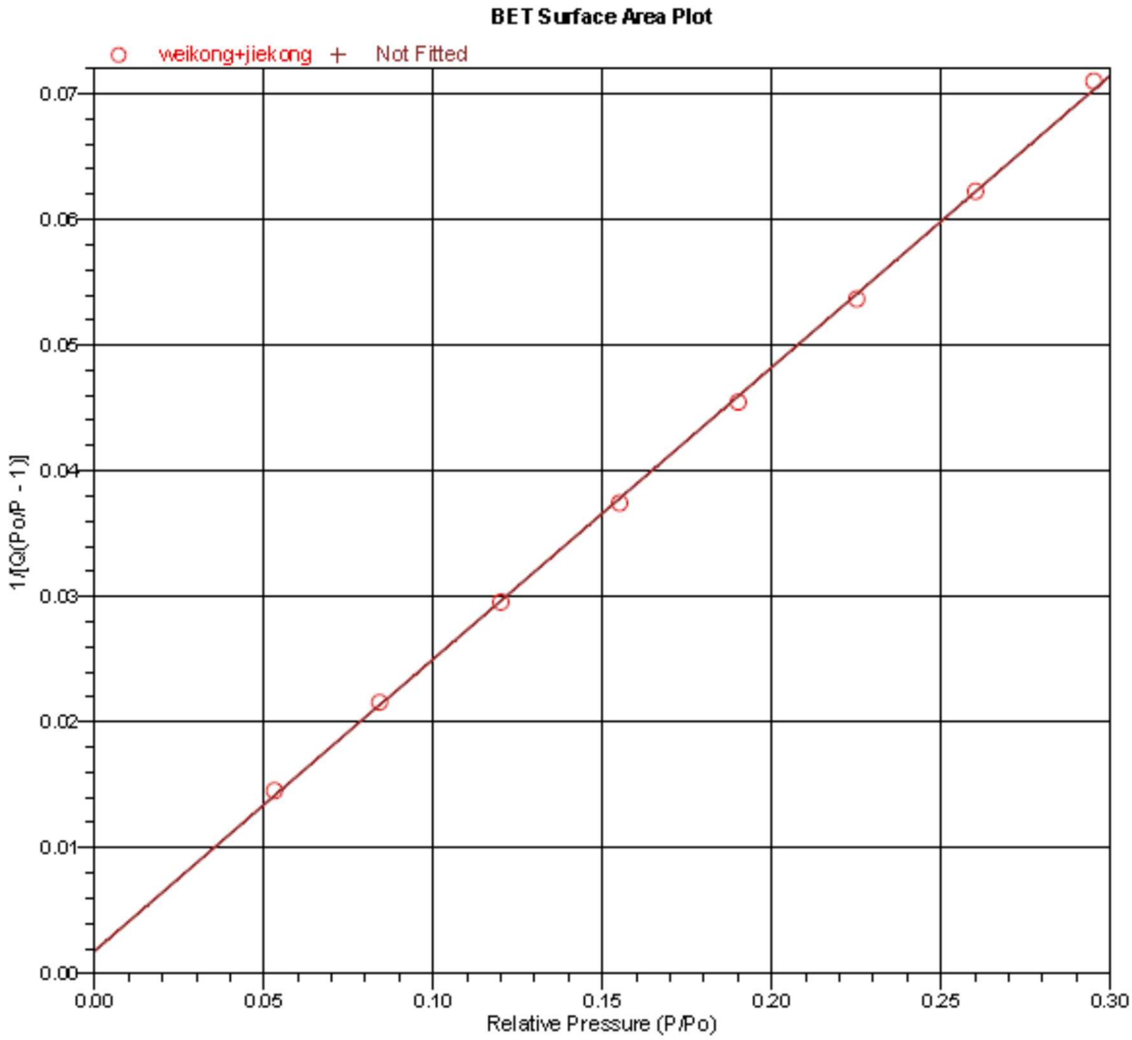

Fig. 10 BET pattern of consolidated sample at 12% of sugar cane molasses.

Figs. 11 and 12 show that the nitrogen adsorption-desorption isotherm on the sample stabilized at 12% sugarcane molasses corresponds to the type IV isotherm. The existence of hysteresis during adsorption-desorption reflects the presence of mesopores.

The opening point (A) and closing point (B) of the hysteresis loop are:

- On the adsorption-desorption isotherm as a function of relative pressure (Fig. 11)

A (10, 40 cm^3/g); B (0.7, 10 cm^3/g)

- on the adsorption-desorption isotherm as a function of absolute pressure (Fig. 12)

A (760 mmHg, 40 cm^3/g); B (545 mmHg, 10 cm^3/g).

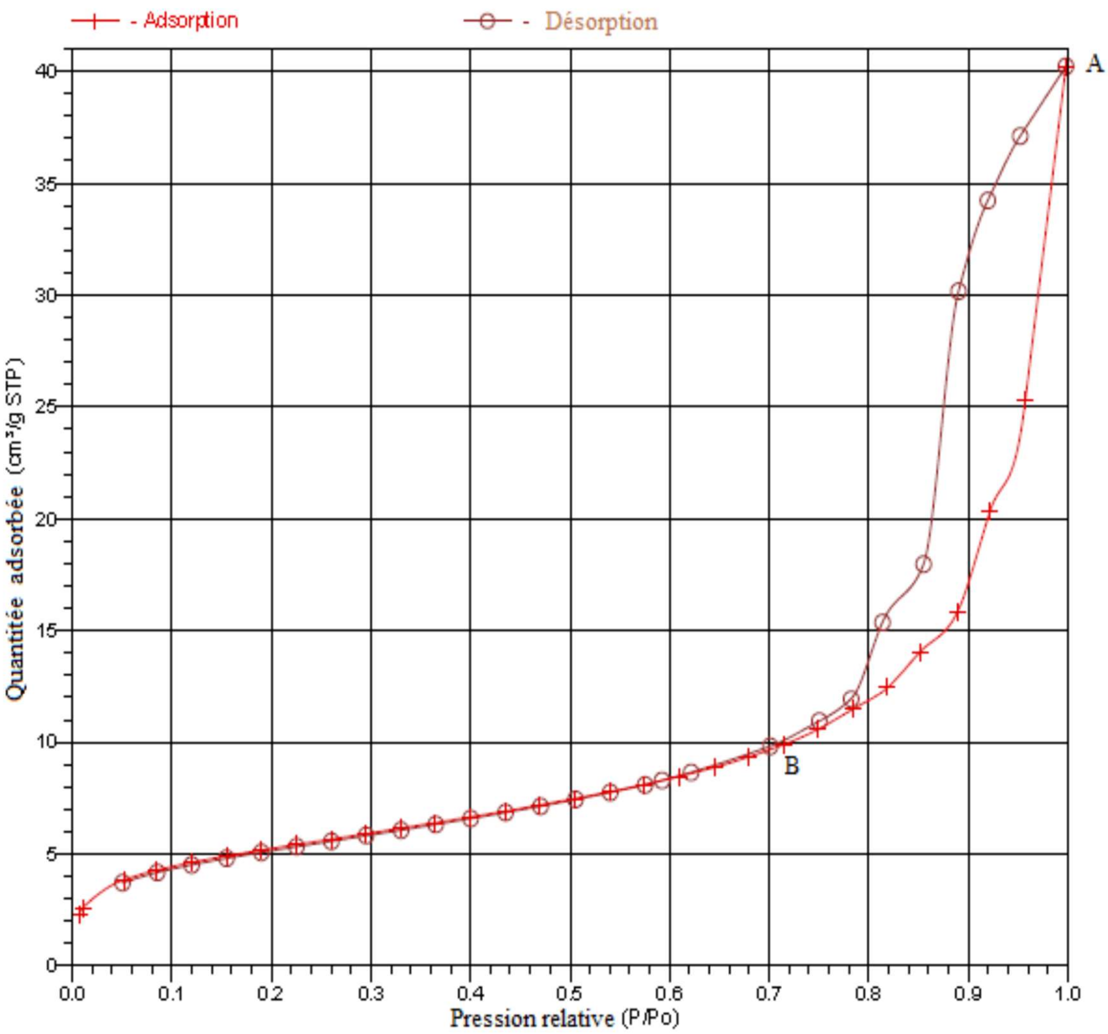

Fig. 11 Adsorption isotherm and nitrogen desorption on consolidated sample at 12% of sugar cane molasses. during relative pressure.

The opening point (A) of the hysteresis loop shows that the 12% molasses stabilized sample adsorbs less gas (40 cm^3/g) than the 0% sample (59 cm^3/g) and adsorbs more gas than the 8% stabilized sample (37 cm^3/g), although all three samples reached the same maximum pressure (760 mmHg). This suggests that the 0% sample is more porous than the 8% and 12% stabilized samples. Nevertheless, the 12% stabilized sample has more accessible porosity than the 8% sample.

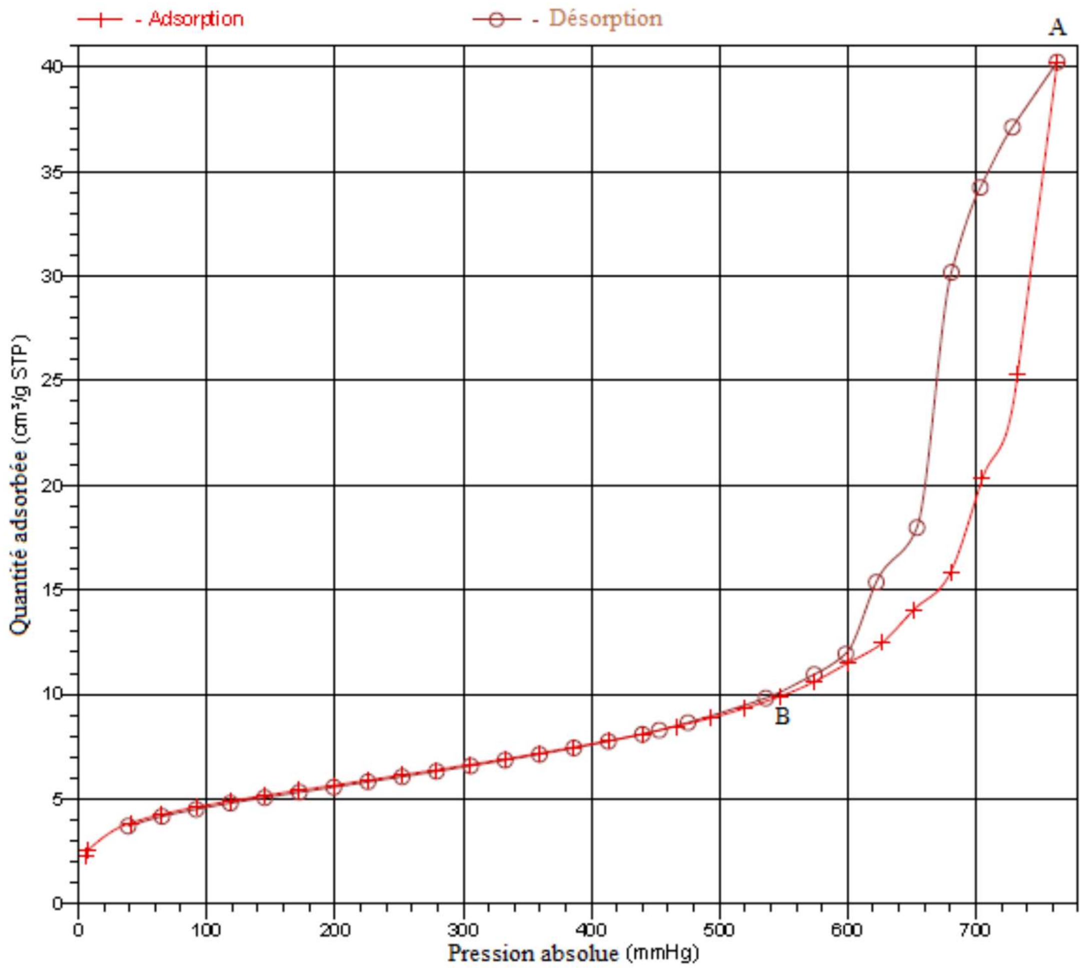

Fig. 12 Adsorption isotherm and nitrogen desorption on consolidated sample at 12% of sugar cane molasses. During the absoluted pressure.

3.4 BET Adsorption Isotherm of Nitrogen on Consolidated Soil at 16% of Sugar Cane Molasses

The obtained experienced results are presented in Table 5.

The isotherm obtained (Fig. 13) using the BET model equation and the calculated parameters are presented in Table 6.

Table 5 Experienced results for the application of BET pattern.

Relative pressure (P/P_0)	Quantity adsorbed (cm^3/g STP)	$1/[Q(P_0/P-1)]$
0.052427469	4.5768	0.012089
0.083587350	5.1039	0.017871
0.119818332	5.5791	0.024400
0.154760759	5.9710	0.030664
0.189839926	6.3338	0.036996
0.224958386	6.6789	0.043458
0.260081362	7.0180	0.050085

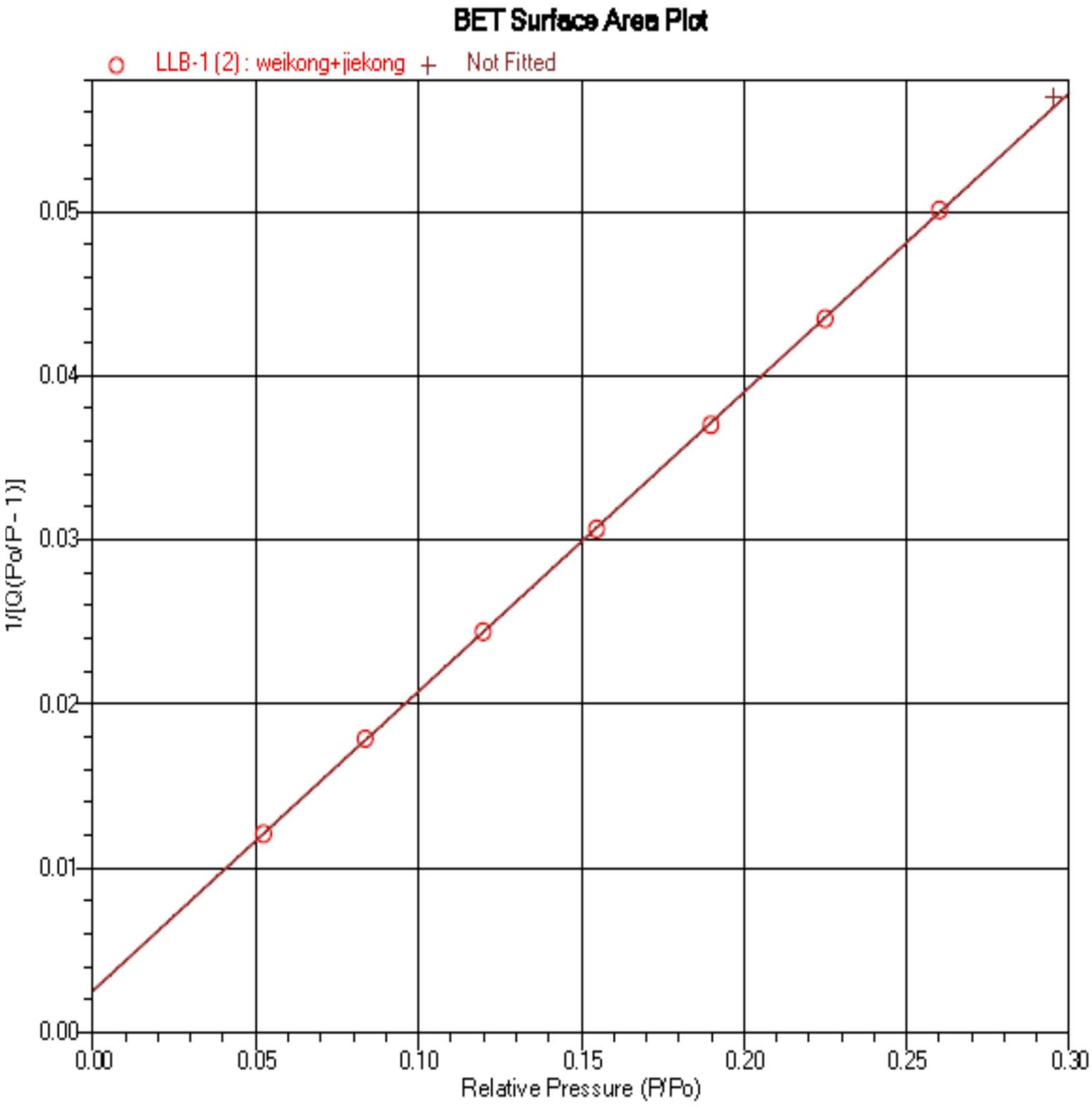

Fig. 13 BET pattern of consolidated sample at 16% of sugar cane molasses.

Figs. 14 and 15 show that the nitrogen adsorption and desorption isotherm on the sample stabilized at 16% sugarcane molasses corresponds to the type IV isotherm which suggests the presence of mesopores.

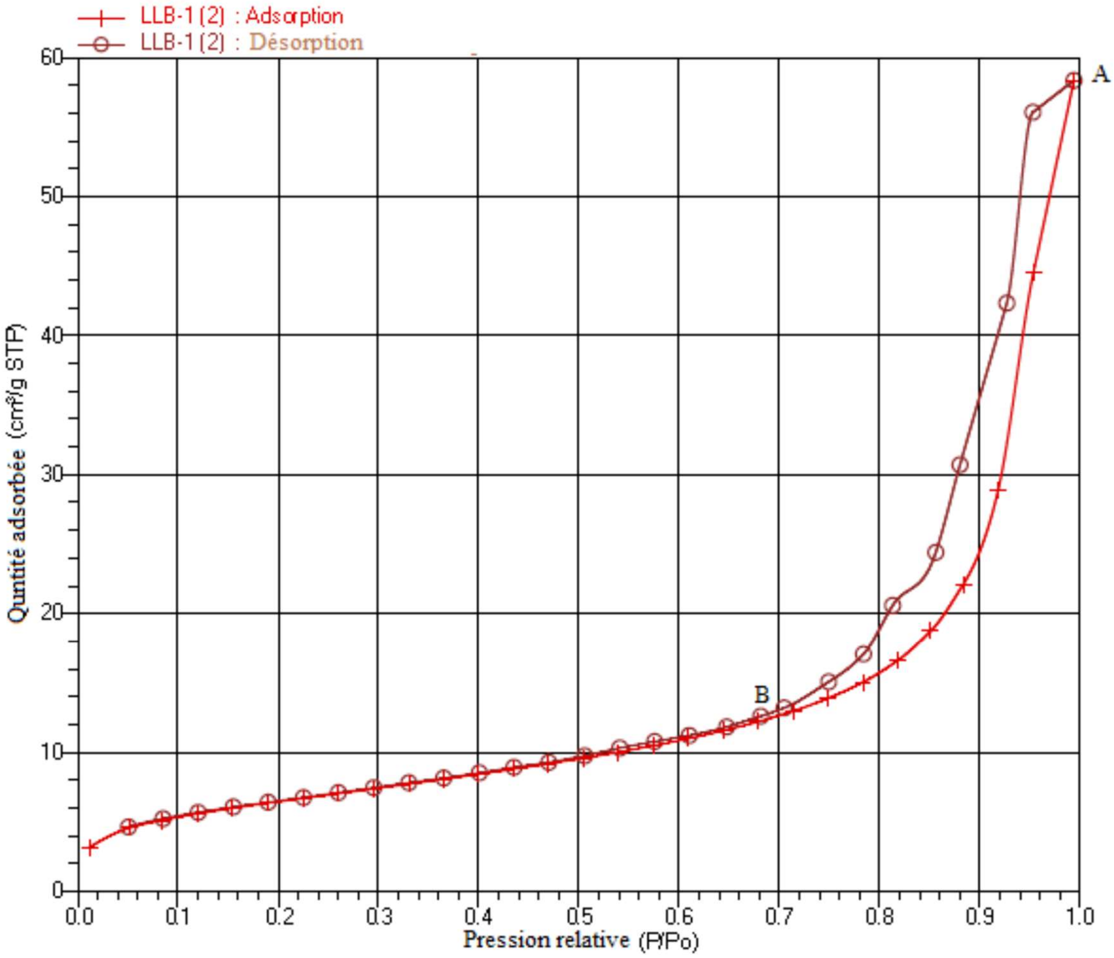

Fig. 14 Adsorption isotherm and nitrogen desorption on consolidated sample at 16% of sugar cane molasses according to relative pressure.

The opening point (A) and closing point (B) of the hysteresis loop are:

- On the adsorption-desorption isotherm as a function of relative pressure (Fig. 14)

A (10, 58 cm^3/g); B (0, 69, 13 cm^3/g)

- on the adsorption-desorption isotherm as a function of absolute pressure (Fig. 15)

A (760 mmHg, 58 cm^3/g); B (520 mmHg, 13 cm^3/g).

The opening point (A) of the hysteresis loop shows that the sample stabilized at 16% molasses adsorbs more gas (58 cm^3/g) than the 8% (37 cm^3/g) and 12% (40 cm^3/g) samples. However, it adsorbs almost the same amount of gas as the 0% stabilized sample (59 cm^3/g), although all four samples reached the same maximum pressure (760 mmHg). This supports the idea that the 16% sample is more porous than the 8% and 12% stabilized samples. But also that the 16% stabilized sample has the same porosity as the 0% sample. These observations are confirmed by the pore volume and pore diameter distribution, micrographs and capillary adsorption porosity.

The hysteresis loop closure points (B) on the isotherms as a function of absolute pressure shows that the closure pressures increase with sugarcane molasses content (at 0% (500 mmHg), 8% (520 mmHg) and 12% (545 mmHg)) and then decreases for the 16% (520 mmHg) content. The hysteresis loops of the adsorption/desorption isotherms of the stabilized soils do not have the same closing point.

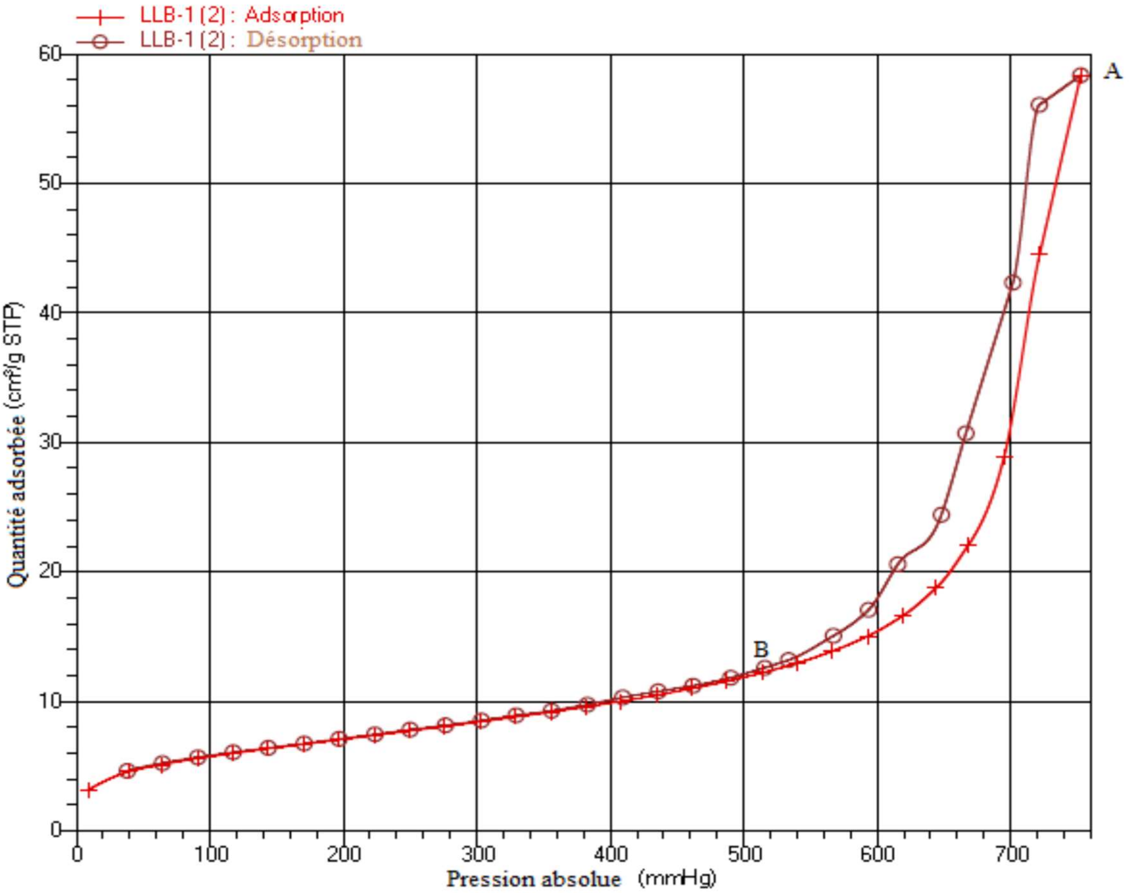

Fig. 15 Adsorption isotherm and nitrogen desorption on consolidated sample at 16% of sugar cane molasses. During the absoluted pressure.

3.5 Comparison of Obtained Parameters from BET Pattern Equation

The obtained isotherm from BET pattern equation and calculated parameters are presented in Table 6.

Table 6 Summary of obtained parameters from BET pattern equation.

	0% of sugar cane molasses	8% of sugar cane molasses	12% of sugar cane molasses	16% of sugar cane molasses
Q_m (cm³/g STP)	7.5457	4.8345	4.2754	5.4135
C	186.141759	295.617810	129.473855	72.591907
Slope (*g*/cm³)	0.131	0.206	0.232	0.182
Y-intercept (*g*/cm³)	0.000712	0.0007	0.0018	0.0025
Sample mass (g)	0.5513	0.4548	0.5307	0.5332
BET area (m²/g)	32.8433	21.0425	18.6091	23.5626
n	112×10^{18}	59×10^{18}	61×10^{18}	78×10^{18}
n'	-	53×10^{18}	51×10^{18}	34×10^{18}

The amount adsorbed required to form a solute monolayer (Qm) decreases with the addition of sugarcane molasses. The sample without sugarcane molasses has the highest Qm value, followed by the 16% sample. The number of gas molecules adsorbed on a monolayer (n) also decreases with the addition of sugarcane molasses. The sample without sugarcane molasses adsorbs more gas on the monolayer than the samples stabilized with sugarcane molasses (8%, 12% and 16%). However, the 16% sample adsorbs more gas on the monolayer than the other samples (8% and 12%).

This result allows us to think that the sample without sugarcane molasses is more porous than the stabilized samples (8%, 12% and 16%). However, the 16% sample is more porous than the others. This result is in agreement with the pore volume and diameter distribution, the micrographs and the porosity by capillary adsorption.

The number of sugarcane molasses molecule adsorbed on the monolayer (n') decreases with the addition of sugarcane molasses. The 8% sample has more sugarcane molasses molecules adsorbed (53.1018) on the monolayer, followed by the 12% sample (51.1018). The 16% sample adsorbs less sugarcane molasses molecule on the monolayer (34.1018) than the other samples (8% and 12%). This suggests that, it is not advantageous to stabilize at 16% sugarcane molasses, because less sugar cane molasses molecule are adsorbed and in addition the porosity is high. Indeed, the addition of molasses as a stabilizer beyond 15% results in a progressive decrease in compressive strength due to the increase in water absorption by the particles [18] [Mahendran and Vignesh (2016)]. The lubrication benefit provided by molasses is negated at higher percentages of additive by dispersion caused by thicker films [19] [Gow (2016)]. The 8% molasses content appears to be the most optimal, as the number of molecules of sugarcane molasses adsorbed is higher and the porosity is less. Gadise Tesema (2015) found this same fact, the strength values (UCS, CBR) of expansive soil are higher at 8% sugarcane molasses [20]. Bizualem Taye (2015), on the other hand confirms that expansive soil treated with 8% sugarcane molasses gives higher CBR values [21]. Prudhvi (2017) reported that the addition of 7% to 10% molasses and lime to expansive soil achieved the optimum strength [22]. Nabeel et al. (2019) attested that molasses was most effective at a percentage of 8%, as beyond that, the dry density of the soil decreased [23]. Mamuye Yibas (2018) stated that the addition of 8% molasses in natural gravel material was suitable for sub-base construction [14].

The shape of the gas adsorption isotherm and hysteresis loop provides information about the pore structures of the material and the gas-solid interactions [Joewondo (2018)] [24]. The shape of the isotherms is the same for all samples and is consistent with both monolayer and multilayer adsorption mechanisms with capillary condensation. Nitrogen adsorption and desorption on the sample without sugarcane molasses and on the samples stabilized with sugarcane molasses shows that the isotherms are all type IV, suggesting the presence of mesopores in all samples and a type H4 hysteresis loop. Nevertheless, not all nitrogen adsorption/desorption isotherms have the same hysteresis loop closure point.

The BET specific surface area, the amount of nitrogen adsorbed on a monolayer (Qm), and the BET constant(C) decreased sharply with increasing amount of sugarcane molasses in the samples. These observations support the idea that the access of nitrogen to the surface of the material is blocked, two reasons seem plausible: the sugarcane molasses inserts into the interstices without bonding to the clay. Or the sugarcane molasses is inserted in the interstices and the organic molecules of the sugarcane molasses establish a bond with the clay. The sugarcane molasses serves as a binder in the latter case. Janekarn et al. (2020) reported that the nitrogen adsorption-desorption isotherms of CSC400 and CSC800 exhibited type IV isotherms. The molasses-stabilized clayey fine soil (kaolinite) has the same characteristics as the cane molasses-doped silicate composites, therefore these materials exhibit the same nitrogen adsorption behavior, i.e., the same type of isotherm (IV) [11]. However, for Sing and Williams (2004), H3 hysteresis loops are typical for N2 adsorption by montmorillonite and lamellar particle aggregates, while H4 loops are observed for carbon isotherms [25]. The soil used in our study being thin and clayey in addition to being reworked contributed to the multiplication of the microporosity which led to the H4 loop, usually obtained for microporous materials like carbons.

4. Conclusion

The results obtained allow us to state the following:

- The addition of sugarcane molasses to the fine clay soil does not change the type of nitrogen adsorption isotherm IV and the type H4 of the hysteresis loop, which justifies the presence of mesopores in the structure of the materials.
- The hysteresis loop closure pressures increase with the sugarcane molasses content. The nitrogen adsorption/desorption isotherms of the stabilized concretes do not have the same closing point on the hysteresis loop.

The maximum amount of gas adsorbed decreases with the addition of sugarcane molasses (8% and 12%) and then increases for the stabilized sample at 16%.

- - The raw earth materials formed using our fine clay soil are mesoporous materials.
- The sugarcane molasses occupies the outer surface of the fine clay soil particles. The 8% stabilized sample adores more (53, 10^8) sugarcane molasses molecules.

References

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