Revista ARACÊ
ISSN: 2358-2472

NANOCLAY PRODUCTION

https://doi.org/10.56238/arev7n8-176

Date of submission: 07/19/2025 **Date of publication:** 08/19/2025

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ABSTRACT
Since primordial times, humanity has used clay as an essential raw material in the commercial and industrial sectors. Nanocomposites such as nanoclay have a significant gain in mechanical properties, being used by industry in various applications. In this article, the raw clay was collected, colloidal suspension was carried out and the nanoclay resulted, with the objective of producing a nanocomposite from the raw clay collected from the soil, presenting its characteristics, chemical compositions and analysis of its structure. The methodology was followed in the production of nanoclay through colloidal suspension and physical analysis of the raw clay. The nanoclay produced was followed by Scanning Electron Microscopy (SEM and SEM), X-ray Energy Dispersive Spectroscopy (EDS) and X-ray Diffraction (XRD) analyses. Based on the results that were obtained, it became possible to obtain satisfactory results with the production of nanoclay in this study, being easy to prepare and with low cost, it is possible to prepare a nanocomposite for various applications.

Keywords: Clay. Polymer. Grain. Clay Minerals.

PRODUÇÃO DE NANOARGILA

RESUMO
Desde os tempos primordiais, a humanidade utiliza a argila como matéria-prima essencial nos setores comercial e industrial. Nanocompósitos como a nanoargila apresentam ganho significativo em propriedades mecânicas, sendo utilizados pela indústria em diversas aplicações. Neste artigo, a argila bruta foi coletada, a suspensão coloidal foi realizada e a nanoargila foi obtida, com o objetivo de produzir um nanocompósito a partir da argila bruta coletada do solo, apresentando suas características, composições químicas e análise de sua estrutura. A metodologia utilizada na produção da nanoargila foi por meio de suspensão coloidal e análise física da argila bruta. A nanoargila produzida foi acompanhada por análises de Microscopia Eletrônica de Varredura (MEV e MEV), Espectroscopia de Dispersão de Energia de Raios-X (EDS) e Difração de Raios-X (DRX). Com base nos resultados obtidos, foi possível obter resultados satisfatórios com a produção da nanoargila neste estudo, sendo de fácil preparo e baixo custo, sendo possível preparar um nanocompósito para diversas aplicações.

Palavras-chave: Argila. Polímero. Grão. Minerais de Argila.

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PRODUCCIÓN DE NANOARCILLA

RESUMEN
Desde tiempos remotos, la humanidad ha utilizado la arcilla como materia prima esencial en los sectores comercial e industrial. Los nanocompuestos, como la nanoarcilla, presentan una mejora significativa en sus propiedades mecánicas y se utilizan en la industria en diversas aplicaciones. En este artículo, se recolectó la arcilla cruda, se realizó una suspensión coloidal y se obtuvo la nanoarcilla. El objetivo es producir un nanocompuesto a partir de la arcilla cruda recolectada del suelo, presentando sus características, composición química y análisis estructural. La metodología empleada para la producción de nanoarcilla se basó en la suspensión coloidal y el análisis físico de la arcilla cruda. La nanoarcilla producida se analizó mediante microscopía electrónica de barrido (MEB), espectroscopia de dispersión de energía de rayos X (EDS) y difracción de rayos X (DRX). Con base en los resultados obtenidos, se obtuvieron resultados satisfactorios en la producción de nanoarcilla en este estudio, ya que su fácil preparación y bajo costo permiten preparar un nanocompuesto para diversas aplicaciones.

Palabras clave: Arcilla. Polímero. Grano. Minerales Arcillosos.

ISSN: 2358-2472

1 INTRODUCTION

Clay is a fine-grained soil consisting of particles with dimensions smaller than 0.002 mm, presenting cohesion and plasticity, according to NBR 6502/95. Thus, it is possible to see that the term clay does not have a mineralogical connotation, being applied to all fine-grained particles found in sediments and soils (MORAIS, 2021).

Most of the properties of clays for a given purpose come from the clay minerals present, which have variations in crystallinity and are accompanied by other non-clayey minerals such as quartz, feldspars, calcite, hematite, mica, among others, with a particle size of 2 μm (MORENO, 2012).

The very versatile materials, abundant in nature, which have extremely fine granulometry, are non-toxic and low cost, are clays (MORAES *et al*., 2009).

Currently, millions of tons of clay find applications, not only in ceramics and construction materials, slurries, foundry molds, pharmaceutical products, but also as adsorbents, catalysts, catalyst support, ion exchangers, among others, depending on the specific properties (MORAES *et al*., 2009).

The term nanoclay is used to designate clay minerals of the group of phyllosilicates that are silicates in sheets, having layer thicknesses of approximately 1 nm and other dimensions in the order of 50 nm to 150 nm and have a specific surface area close to 750 m^2/g, the specific surface area of clean sand is approximately 0.0002 m^2/g, while Portland cement has a specific surface area between 0.30-0.35 m^2/g (CARASTAN, 2007).

This chapter aims to present the production of nanoclay from raw clay collected from the soil, presenting its characteristics, chemical compositions and analysis of its structure.

2 METHODOLOGY

2.1 OBTAINING THE CLAY

The kaolinite clay found in the Amazon region, Acre, was collected and the process to produce nanoclay was carried out. The collection took place in the Industrial District located in Rio Branco/AC, with the following coordinates S 9º 56' 33'', W 67º 52' 11'', see figure 1.

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Figure 1

Clay collection site.

Source: Google Maps (2023).

2.2 PHYSICAL CHARACTERIZATIONS OF CLAY

For the raw clay, physical analyses were carried out, such as: the particle size by screening according to the DNER-ME 080/94 standard, Atterberg limits with the determination of the plasticity limit through the DNER-ME 082/94 standard and the determination of the liquidity limit according to the DNER-ME 122/94 standard and the actual density according to the DNER-ME 093/94 standard.

Before carrying out the analyses, the soil was prepared for characterization tests using the DNER-ME 041/94 standard. All analyses were carried out at the Soil Laboratory of the Federal University of Acre.

2.3 PRODUCTION OF NANOCLAY

After collecting the raw clay, it was dried in the open air for 24 hours and unearthed, then sieved in sieve No. 200, the sieved material was used to produce nanoclay following the methodology adapted from Araújo (2019). The clay was prepared with a colloidal suspension through the process: with the concentration of 300 g to 2000 mL of distilled water and placed under stirring for 30 minutes. Then ultrasonically by the Ultrasonic Processors for 8 minutes following an eight-fold sequence, the resulting solution was kept at rest for one week. After one week, the supernatant part of the suspension was removed and it was placed under agitation for a period of 12 h, after which the suspension was centrifuged at 4000 rpm for 20 m, to remove the precipitate and eliminate any remaining clay aggregate. Finally, this colloidal suspension of aggregate-free clay was dried for 48 h at 45 ºC in an oven where the nanoclay powder was obtained.

The productions and materials used for nanoclay were carried out at the Nanobiotechnology Laboratory of the Bionorte Complex of the Federal University of Acre.

2.4 SCANNING ELECTRON MICROSCOPY (MEV AND SEM) AND X-RAY ENERGY DISPERSIVE SPECTROSCOPY (EDS)

The nanoclay sample with the MEV and SEM, were used to inspect the microstructures of the materials under different conditions and the EDS analysis to obtain the chemical composition. All analyses were performed with a Thermo Axia ChemSEM (Thermo Fisher Scientific), operated at an acceleration voltage of 10 kV, under low vacuum conditions (50 Pa). The analyses took place at the University of Illinois-USA, in the laboratory of the department of materials science.

2.5 X-RAY DIFFRACTION - XRD

The X-ray diffraction (XRD) patterns of the nanoclay samples were measured using a diffractometer (BRUKER D8-ADVANCE). The XRD measurements conditions performed between 10º-80º at 2θ, at an acceleration voltage of 40 kV and beam current of 40 mA, using step sizes of 0.02º and a scan speed of 0.1 sec/step. The analyses took place at the University of Illinois-USA, in the laboratory of the department of materials science.

3 RESULTS AND DISCUSSION

The results are presented below, from obtaining the raw material used to the result of the material produced.

3.1 OBTAINING THE CLAY

It was collected to carry out the process of obtaining nanoclay, the clay found in the Amazon region, Acre. The collection took place in the Industrial District located in Rio Branco/AC, as shown in figure 2.

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Figure 2

Place of collection of the clay used.

Source: From the authors (2023).

The location shown in figure 2 was chosen because there is a concentration of clay already removed from the deeper soil for application in ceramics.

3.2 PHYSICAL CHARACTERIZATIONS OF CLAY

With the raw clay collected, physical analyses were carried out on the clay such as sieving granulometry, the results are presented in Chart 1.

Chart 1

Particle size analysis by sieving.

PARTICLE SIZE ANALYSIS BY SCREENING						
AIR-DRIED SOIL SAMPLE		HYDROSCOPIC MOISTURE			SUMMARY %	
Wet total sample	1960.9	Capsule No.	13	41	Boulder above 4.8 mm	0.02
Retained #No. 10 dry	14.17	C+S+A	87.8	104.5	A. Thick 4.8 - 2.0 mm	0.72
Retained #No. 10 wet	1946.73	C+S	85.73	102.16	A. Average 2.0 - 0.42 mm	2.37
Water weight	60.35	A = water	2.07	2.34	A. Fine 0.42 – 200 mm	10.37
Passes # No. 10 dry	1886.38	C = capsule	20.58	28.41	Passing #200	86.52
Wet < No. 10 Sample	200	S = soil	65.15	73.75	Total	100
Dry < No. 10 Sample	193.8	H = Humidity	3.2	3.2	Retained at #200	

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Dry total sample	1900.55	Hm =	3.2	Fc = 0.969		
SIEVE (NO.)	RETAINED MATERIAL					% that exceeds the total sample
	Weight (g)	Sample % Smallest No. 10	% of total sample	Cumulative %		
4	0.39		0.02	0.02		99.98
10	13.78		0.72	0.74		99.26
30	2.76	1.42		1.42	98.58	97.85
40	1.89	0.97		2.39	97.61	96.89
80	9.11	4.7		7.09	92.91	92.22
200	11.12	5.74		12.83	86.52	86.52

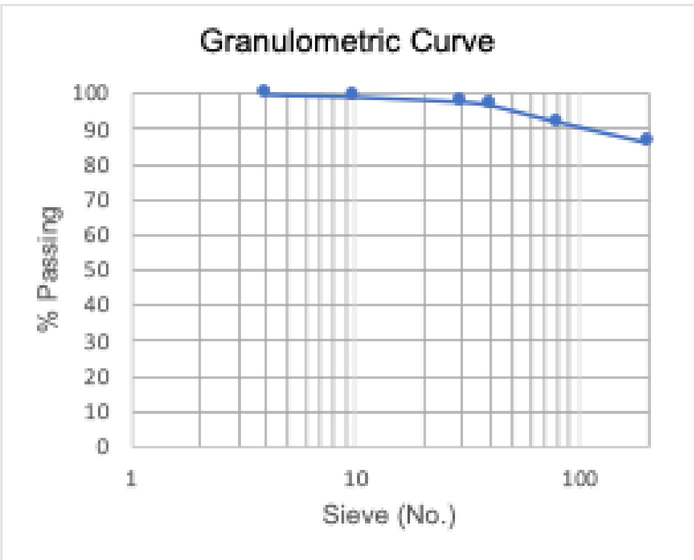
Source: From the authors (2023).

It is observed in the results of the sample that for the boulder they were 0.02 mm, which means that in the analysis there was no gravel, because it needed to be above 4.8 mm, for coarse sand it was 0.72 mm, not being in the range of 4.8 mm and 2.0 mm, but there is medium sand with a value of 2.37 mm and fine sand with 10.37 mm.

Graph 1 shows the particle size curve made with sieves no. 4, 10, 30, 40, 80 and 200.

Graph 1

Particle size curve.



Source: From the authors (2023).

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The dimensions of aggregates were observed as a function of the size of the rock fragment and its proper particle size classification, being 10.37 % of fine sand, 4.37 % of medium sand, 0.72 % of coarse sand and 0.02 % of gravel.

The particle size curve presents the graphic results of the retained percentages accumulated in each sieve in relation to the dimension of the opening of its mesh, the accumulated retained percentage is represented on a natural scale and the sieve opening on a logarithmic scale.

The Atterberg Limits were performed on the raw clay, the results are presented in Table 2, and to achieve them a series of tests and trials were carried out to define the Liquidity Limit, the Plasticity Limit.

Chart 2

Atterberg's limits.

ATTERBERG LIMITS							
LIQUIDITY LIMIT							
Capsule No.	Capsule + Wet Soil (g)	Capsule + dry soil (g)	Capsule (g)	Water (g)	Dry soil (g)	Moisture content (%)	Number of Strikes
42	18.09	14.32	5.95	3.77	8.37	45.04	52
23	18.95	14.64	5.44	4.31	9.2	46.85	42
20	20.01	15.32	5.92	4.69	9.4	49.89	31
15	19.55	14.64	5.5	4.91	9.14	53.72	20
11	20.87	15.3	5.76	5.57	9.54	58.38	10
PLASTICITY LIMIT							
Capsule No.	Capsule + Wet Soil (g)	Capsule + dry soil (g)	Capsule (g)	Water (g)	Dry soil (g)	Moisture content (%)	Plasticity limit (%)
5	11.71	10.64	6.21	1.07	4.43	24.15	24.49
57	13.66	12.56	7.98	1.1	4.58	24.02	
16	10.22	9.36	5.8	0.86	3.56	24.16	
49	13.59	12.5	8.12	1.09	4.38	24.88	
18	12.22	10.86	5.47	1.36	5.39	25.23	
FINDINGS							
LIQUIDITY LIMIT %					51.8		
PLASTICITY LIMIT %					24.49		
PLASTICITY INDEX %					27.31		

Source: From the authors (2023).

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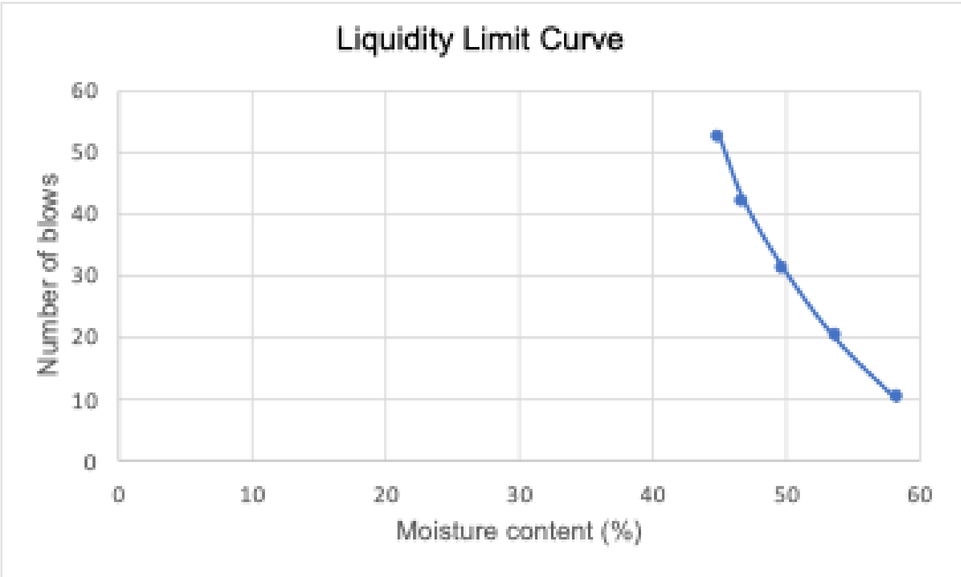
It was observed that the liquidity limit was 51.8%, plasticity limit was 24.49% and the plasticity index was 27.31%. The Plasticity Limit was defined as the lowest moisture content with which a cylinder with a diameter of 3 mm was able to be molded, rolling the soil with the palm of the hand, and the Liquidity Limit marks the transition from the semi-plastic state to the plastic state.

Graph 2 presents the liquidity curve according to the number of strokes in relation to moisture content.

Graph 2

Liquidity Limit Curve.



Source: From the authors (2023).

With the results, it was observed that with the points a straight line is obtained, with this the result that the soil is clayey with the classification of HRB A7-5 is high.

The actual density of the raw clay was analyzed to verify the relationship between the specific weight of the solid particles in grams (g), and the specific weight of an equal volume of pure water at a temperature of 25 ºC, the results are presented in Table 3.

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Chart 3

Actual specific mass of soil grains.

ACTUAL SPECIFIC MASS OF SOIL GRAINS							
Pycno meter No.	Pycnometer + Water (g)	Pycnometer + Soil (g)	Pycnometer + Soil + Water (g)	Empty pycnometer (g)	Temperature ºC	Actual specific mass (g/cmˆ3)	Average specific mass (g/cmˆ3)
1	158.84	84.36	171.58	64.38	25	2.756	2.786
3	163.07	85.13	175.97	65.14	25	2.816	

Source: From the authors (2023).

The relative density of the particles that make up the soil was on average 2.786 g/cm3, where it is evaluated that the real behavior of the volume of the solid in the analysis allowed to disregard the spaces occupied by water and air. The result represents that the actual density is quite stable, being influenced by the composition of the solid fraction, especially in relation to the content of organic matter and mineralogical constituents of the soil, ranging from 2.3 g/cm3 to 2.9 g/cm3.

Frankievicz and Bazzotti (2012) state that the Real Density (Dr) or Particle Density (Dp) corresponds to the real volume that a given solid occupies, not considering its porosity, where the results are expressed in grams per cubic centimeter and its variation is around 2.3 to 2.9 g/cm³.

3.3 PRODUCTION OF NANOCLAY

The clay was collected to make the nanoclay, first the raw clay was prepared, figure 3 shows the drying and unraveling.

Figure 3

Drying and unraveling of the raw clay - a) clay for drying; b) unraveling of the raw clay.

Source: From the authors (2023).

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It can be seen in figure 3 (a) that the drying was carried out in the open air, then, in figure 3 (b), that the unraveling was carried out.

Figure 4 shows the sieving carried out after unraveling the sieved material was to produce the nanoclay following the methodology.

Figure 4

Sieved clay - a) screening; b) sifted clay.

Source: From the authors (2023).

The clay was prepared by colloidal suspension with agitation and ultrasound of the clay concentration, as can be seen in figure 5.

Figure 5

Agitation and ultrasound in the preparation of nanoclay - a) agitation concentration; b) Concentration in Ultrasonic Processors.

Source: From the authors (2023).

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According to figure 5 (a), the clay concentration was performed, then in figure 5 (b) the ultrasound was performed.

To define clays, Reis (2018) states that particle dimensions are the important parameter, in geology the term clay is given to a rock composed of at least 50% of the material with particles smaller than 2 μm, for engineering and soil science the common for the dimension is less than 4 μm, and for the science of colloids the value is less than 1 μm.

Figure 6 shows the solution resulting from the previous process, where it was kept at rest for a week.

Figure 6

Resulting solution - a) resulting solution before resting; b) resulting solution before resting.

Source: From the authors (2023).

It can be seen in figure 6 (a) that the solution after ultrasound and before resting for one week, the clay remained at the bottom with a volume of 600 mL, after one week the separation of the supernatant was observed and resulted in the nanoclay concentrated at the bottom with a volume of 500 mL as shown in figure 6 (b).

After one week, the supernatant part of the suspension was removed and it was brought under agitation, after which the suspension was centrifuged as shown in figure 7.

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Figure 7

Nanoclay for centrifugation.

Source: From the authors (2023).

It is observed that the centrifugation process was for the removal of the precipitate and elimination of any remaining clay aggregate. Finally, this colloidal suspension of nanoclay free of aggregate was dried in an oven, where the powder was obtained as shown in figure 8.

Figure 8

Nanoclay powder.

Source: From the authors (2023).

The nanoclay resulting from the production is ready for technological applications due to its efficiency and reinforcement, as it has a large surface area and the aspect ratio of the nanoclays brings greater efficiency and reinforcement.

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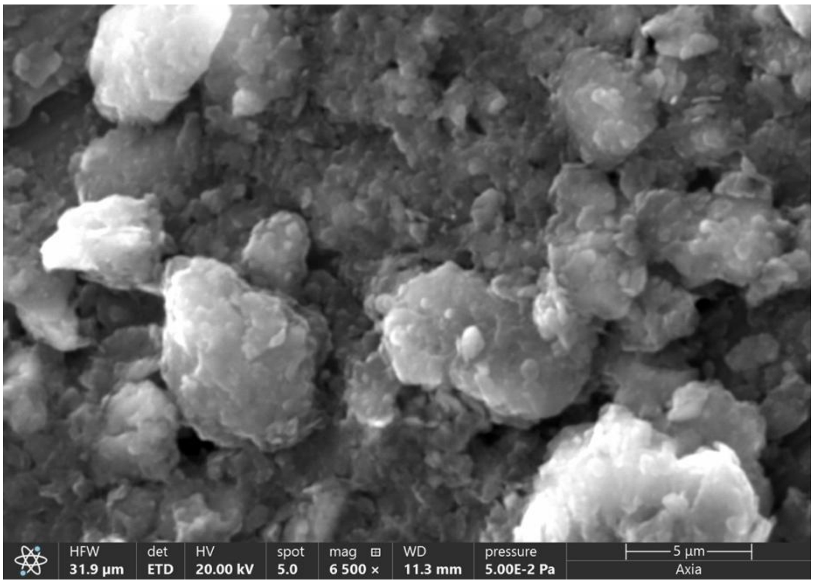
3.4 SCANNING ELECTRON MICROSCOPY (MEV AND SEM) AND ENERGY DISPERSIVE X-RAY SPECTROSCOPY (EDS)

MEV analysis was performed to obtain information on the nanoclay surfaces, according to the equipment and under different conditions and using the 5 μm measurement. It is observed in figure 9, the MEV analysis of the nanoclay that has a homogeneous morphology with uniform pores, compact and aggregated, this aggregate is related to the presence of iron (Fe), that is, they are typical characteristics of stacked lamellar structures with low surface porosity.

Figure 9

MEV nanoclay.



Source: From the authors (2024).

Similar morphologies were found in the study by Lima Junior (2024), who states that stacked lamellar structures are common in nanoclays that have a smooth, continuous and compact morphology, and the perception of the surface roughness that forms agglomerates is due to the presence of iron.

The SEM analysis also provides information on the surfaces of the nanoclay according to the equipment and under different conditions, using the 5 μm measurement. According to the results, it is observed in figure 10 the SEM analysis of the nanoclay, it has a compact, morphologically aggregated sample and voluminous regions.

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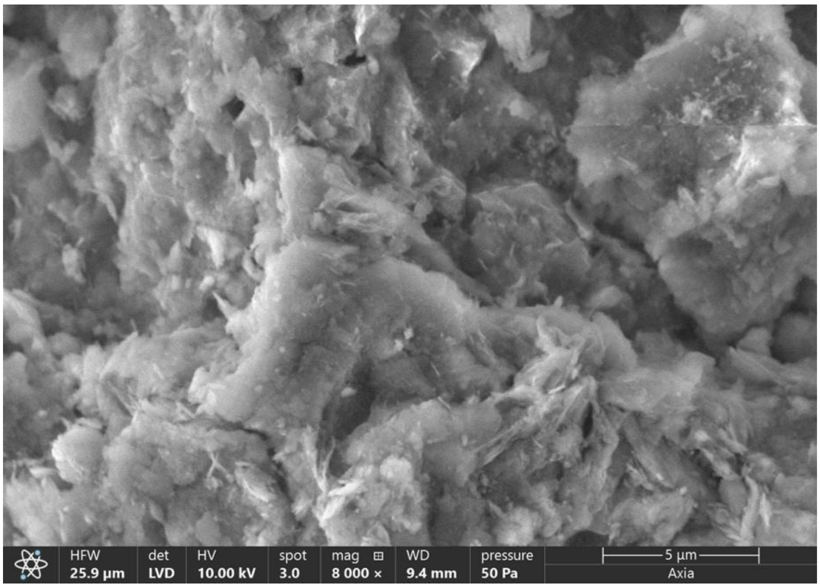
Figure 10

SEM nanoclay.

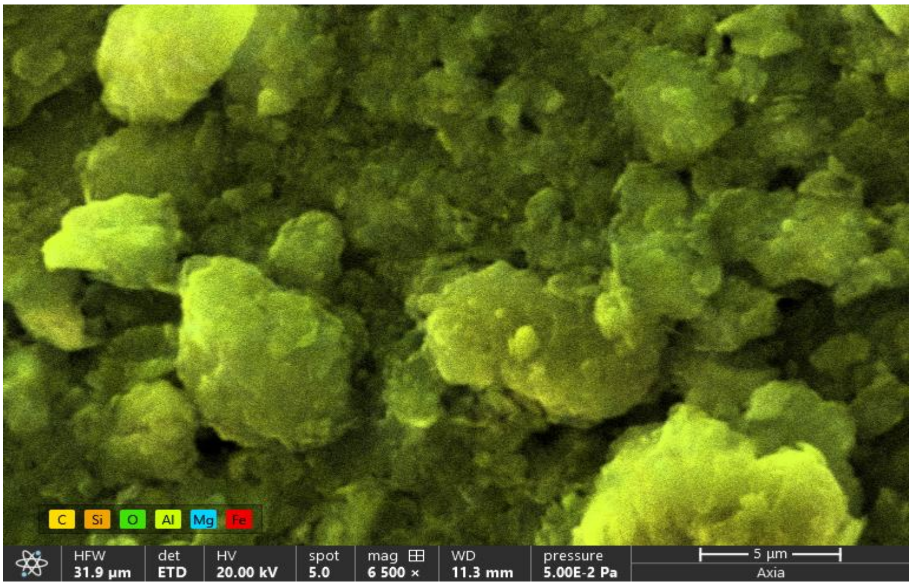


Source: From the authors (2024).

To confirm the presence of chemical components in the nanoclay sample, EDS analysis was performed and coupled to the SEM. Figure 11 shows that nanoclay has the following chemical components, Carbon (C), Silicon (Si), Oxygen (O), Aluminum (Al), Magnesium (Mg) and Iron (Fe).

Figure 11

EDS in nanoclay.



Source: From the authors (2024).

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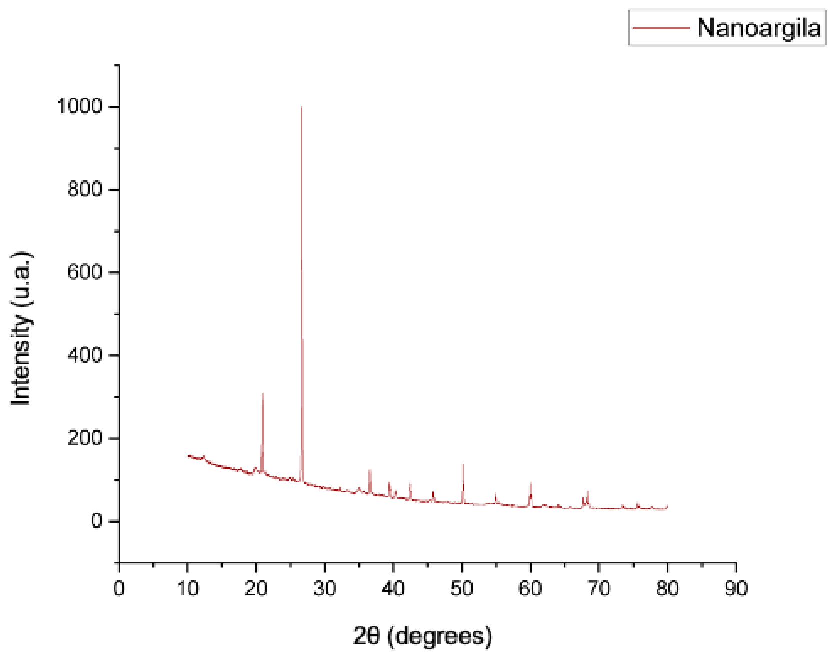
Ferreira Junior (2015) in his study, states that for pure nanoclay, the presence of oxygen with 60% mass or 72.0% atomic, silicon in 27% mass or 18.5% atomic, aluminum with 9% mass or 6.4% atomic, sodium in 2.6% mass or 2.2% atomic and manganese with 1.4% mass or 1.1% atomic, was detected. There was also an increase in the concentrations of nanoclay ions containing 20 % in its structure.

3.5 X-RAY DIFFRACTION - XRD

The crystalline phase of the nanoclay was detected in X-ray diffraction (XRD), analyzing the intensity by the degree. Figure 12 shows the two characteristic peaks at 2 θ of 20.88º and 26.66º, these peaks found are characteristic of the presence of quartz, a common mineral found in nanoclays and the presence of aluminum constitutes the 26.66º peak.

Figure 12

XRD of the nanoclay.



Source: From the authors (2024).

Values of similar peaks were found by Ferreira Junior (2015), in his study the diffractogram of pure nanoclay has peaks of 2 θ equal to 19.9 º, 21.9 º and 28.3 º, related to the mineral present in the nanoclays called quartz and aluminum that is also present in the samples.

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4 CONCLUSION

It can be concluded that the raw clay collected from the soil and passed through soil characterization and preparation processes, obtained the nanoclay ready for technological applications that has efficiency and reinforcement.

The nanoclay produced is a nanostructured material with several nanotechnological applications in materials engineering and civil engineering and can be efficiently applied in industrialized mortar such as adhesive mortar.

The dimensions of the aggregates were observed as a function of the size of the rock fragment and its proper granulometric classification, being 10.37 % of fine sand, 4.37 % of medium sand, 0.72 % of coarse sand and 0.02 % of gravel before the formation of the nanoclay, results from the raw clay.

With the SEM analysis of the nanoclay it was possible to see a homogeneous morphology with uniform pores, compact and aggregated, this aggregate is related to the presence of iron (Fe), that is, they are typical characteristics of lamellar structures stacked with low surface porosity.

According to the results, with the SEM analysis it is concluded that the nanoclay has a compact, morphologically aggregated sample and voluminous regions. The EDS of the nanoclay found the following chemical components, Carbon (C), Silicon (Si), Oxygen (O), Aluminum (Al), Magnesium (Mg) and Iron (Fe).

Finally, with XRD, two characteristic peaks at 2 θ were found in the nanoclay, of 20.88 º and 26.66 º, these peaks found are characteristic of the presence of quartz, a common mineral found in nanoclays and the presence of aluminum constitutes the peak 26.66 º.

It is concluded with the production of nanoclay in this study, being easy to prepare and with low cost, a nanocomposite can be prepared for various applications.

ACKNOWLEDGMENT

The authors' thanks go to the Nanobiotechnology Laboratory for providing the materials and place to carry out the research, to the Graduate Program of the Biodiversity and Biotechnology Network of the Legal Amazon and to CAPES.

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