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Bentonites from Boa Vista, Brazil: Physical, Mineralogical and Rheological Properties

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The aim of this work is to characterize physically and mineralogically six samples of natural and industrialized bentonites from Paraíba, Brazil, and to study its rheological properties to be used as a components of water based drilling fluids. Also it is intended to compare the evolution of the mineralogical composition and rheology of these clays after 40 years of exploitation. The natural bentonite clays were transformed into sodium bentonite by addition of concentrated Na_2CO_3 solution. The suspensions were prepared with 4.86% w/w to measure their rheological properties (apparent and plastic viscosities and water loss). The results showed that: i) the samples present typical mineralogical compositions of bentonites, but after four decades of exploitation, presents inferior quality and ii) among the clays samples, only one presented satisfactory rheological properties be used as a components of water based drilling fluids.

Keyword: *Bentonite, mineralogical characterization, rheology, drilling fluids*

1. Introduction

The name bentonite was suggested initially to the plastic clays found in Fort Benton, Wyoming, USA. These clays present the property of increasing several times its initial volume in the presence of humidity. They are composed, predominantly, of smectite clay minerals, and it is usually sodium and calcium montmorillonites[1].

The bentonites are included in the class of the minerals with larger industrial interest. According to Murray[2], the several industrial applications of these clays are due to its physical and chemical properties (such as, high surface area and cation exchange capacity). They are traditionally applied in oil well drilling mud, as a bonding agent for foundry molding sands, pelletizing iron ores, sealants, animal feed bonds, bleaching clay, agricultural carriers, pet litter adsorbents, adhesives, pharmaceuticals, emulsion stabilizers, desiccants, catalysts, cosmetics and paint, and, recently, it has been used in nanocomposites. The importance of these practical applications is related to rheological properties[3]. Sodium montmorillonite clay is particularly of interest due to its high swelling capacity and formation of a gel-like structure at relatively low clay concentrations[4].

Research performed in the early 90's by the Bureau of Mines of the U.S.A, showed that the sum of bentonite deposits in the world is about 1.36 billion tons, and the U.S.A. has more than 50.0% of the total[5]. In Brazil, the bentonites can be found in relatively small deposits. The most important one is located in the northeast of Brazil, Paraíba State, in the city of Boa Vista (Fig. 1). These clays were discovered in the beginning of the 60's and the first studies showed that these clays were naturally polycationic and could be transformed into sodium bentonites with rheological properties in accordance with the specifications of American Petroleum Institute - API. It was verified that these clays were composed of the clay minerals montmorillonite, illite, kaolinite and mixed layers of illite-montmorillonite and quartz. Also it was established that when these clays are treated with a concentrated sodium carbonate solution it is possible to obtain sodium bentonites capable to substitute the natural sodium bentonites imported by Brazil[6]. An extensive study related with the treatment of these clays with sodium carbonate was done, and resulted in 18 different processes[7].

In the 70's and 80's decades research was carried out in the Federal University of the Paraíba-UFPB[8-14] and in the University of São Paulo-USP[15,16] with the objective to develop techniques of ionic exchange treatment to improve the rheological properties of these clays.

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The origin of the clays from Boa Vista-PB was studied by Gopinath *et al.*[17] and showed that these clays are alterations of glassy material, derived from volcanic ash. Years later, in 1988, the same authors showed that these clays are composed of montmorillonite, illite, kaolinite, quartz and feldspar and can be labeled bentonite[18].

With the discovery of these bentonites Brazil gradually started to produce bentonites in the sodium and calcium forms, supplying the demands of the internal market. According to the National Department of Mineral Production - DNPM, the amount of natural and treated (sodium bentonite) clays produced in the Paraíba State represents 96.0% of the national production and 74.0% of this amount is produced in Campina Grande City, Paraiba State and 4.0% is produced in the São Paulo State[5].

The drilling fluids, also called muds, are used in the oil wells drilling process to remove cuttings, to keep formation fluids confined to their formations, to lubricate the bit, and to build an impervious coating on the wall of the hole in order to impede the penetration of water from the drilling fluid into the formation[1]. According to Caenn and Chillingar[19], the water based drilling fluids are used all over the world, in most of the perforations. *Petrobras*, the Brazilian Petroleum Company, uses water based drilling fluids with bentonite clay in onshore and offshore perforations. Almost all of this clay comes from Boa Vista City, Paraíba State.

Nowadays, after 40 years of exploitation some different type of clays from Boa Vista, PB become exhausted and others become rare, such as *Chocolate* clay having good properties. On the other hand there are some clays found in great amount such as *Bofe* e *Verde-lodo* but with inferior quality.

The objectives of this work are: i) to characterize physically and mineralogically three samples of natural bentonites from Boa Vista City, Brazil and three industrialized samples; ii) to study the rheological properties of these clays to be used as thixotropic agents for water based drilling fluids and iii) to carry out a comparative study of the evolution of the mineralogical composition and rheology of these clays after 40 years of exploitation.

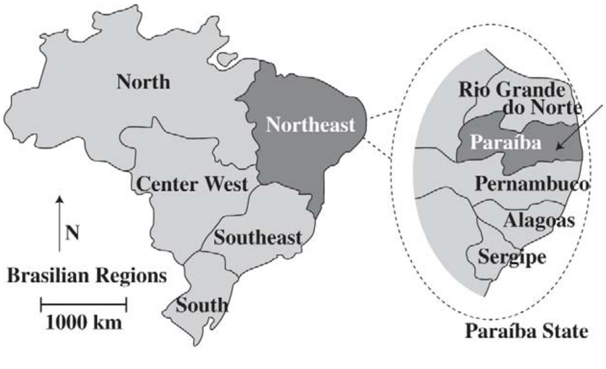


Figure 1. Location of the studied area.

2. Materials and Methods

2.1. Bentonites

Three samples of natural bentonites were studied, from Boa Vista City, Paraíba State, Brazil, namely locally as *Bofe*, *Chocolate* and *Verde-lodo*. Three samples of industrialized bentonites in the sodium form, supplied by local industries and identified as *Dolomil*, *Brasgel* and *Brasgel PA*, were also studied. The natural clays were dried at 60 ± 2 °C for a period of 7 days, milled and screened in a ASTM 200 (aperture size of 0.074 mm) sieve.

2.2. Physical and mineralogical characterization

The water content and the particle size were evaluated according to the Brazilian norm N-2605[20]. The cation exchange capacity (CEC) and the surface area were determined by the method of methylene blue adsorption[21].

The chemical composition of the clays was analyzed according to the methods developed by the Laboratory of Mineral Analyses (LAM), Center of Sciences and Technology (CCT), Federal University of Campina Grande (UFCG), Brazil[22].

Differential thermal analysis (DTA) and thermogravimetric analysis (TGA) were obtained with a BP Engenharia equipment, model BP 3000, operating at a rate of 12.5 °C/min and maximum temperature of 1000 °C. For DTA calcined aluminum oxide (Al_2O_3) was used as a reference powder.

The X-ray diffraction pattern of the natural and industrialized samples and treated with ethylene glycol were obtained with a Diffractometer Siemens/Brucker, model AXS D5005, with CuKα radiation ($\lambda = 1.54056$ Å).

Infrared spectrums were obtained with a Nicolet Avatan equipment, model 360, operating in the range of 4000-400 cm^{-1}. The sample powders were tested in the form of a disc by pressing using potassium bromide (KBr).

Transmission electron micrographs were obtained with a TEM Philips CM 200 equipment, operating at 200 kV.

2.3. Transformation of natural bentonites into sodium bentonites

The natural clays were treated with Na_2CO_3 concentrated solution (200 g/L) in proportions of: 75, 100, 125, 150 and 175 meq/100 g of dry clay for the sample *Bofe*, and 50, 75, 100, 125 and 150 meq/100 g of dry clay for the sample *Chocolate* and *Verde-lodo*. After, the samples were cured for a period of 5 days in humid chamber, according to the process developed by Zandonadi *et al.*[7].

2.4. Preparation of suspensions

The suspensions of the natural clays treated with a Na_2CO_3 solution and the industrialized clays in a concentration of 4.86% w/w were prepared according to the

Petrobras standards N-2605[20]. After, the suspensions remain for a period of 24 h in a humid chamber, at 100% of relative humidity.

2.5. Determination of the rheological properties

The rheological properties of the suspensions were carried out according to the *Petrobras* standards N-2605[20]. The apparent viscosity (AV) and plastic viscosity (PV), were measured by using a Fann viscosimeter, model 35A, and the water-loss (WL) through a filter paper, was determined by using a filter press Fann model. The pH was evaluated by using a digital pH meter, Analyser model.

Table 1. Water content and particle size for the natural and industrialized bentonites.

Samples	Water content (%)	Particle size + 200 Mesh (0.074 mm) (%)
Bofe	2.77	0.02
Chocolate	4.31	0.44
Verde-lodo	2.45	0.06
Dolomil	3.79	2.00
Brasgel	2.88	0.10
Brasgel PA	5.28	1.26
Maximum value*	14.0	4.0

* According to the *Petrobras* specification[23].

Table 2. Cation exchange capacity (CEC) and surface area determined by the method of methylene blue adsorption.

Samples	CEC (meq/100 g of dry clay)	Surface area (m^2/g)
Bofe	76	593
Chocolate	92	717
Verde-lodo	68	530
Dolomil	100	780
Brasgel	104	811
Brasgel PA	104	811

3. Results and Analysis

3.1. Physical and mineralogical characterization

3.1.1. Water content and particle size

According to the *Petrobras* specification N-2604[23], the water content of the natural and activated clays should not exceed the maximum of 14.0%. Through the data contained in the Table 1, it is observed that all the samples presented water contents below the maximum, with exception of the samples *Chocolate* and *Brasgel PA*.

In relation to the particle size, the samples *Bofe* and *Verde-lodo* contained the smallest amount of coarser grains (> 74 µm). According to the *Petrobras* standards N-2604[23], all samples contained particle sizes below the specification (< 4.0%).

3.1.2. Cation exchange capacity and surface area

The values of the cation exchange capacity ranged from 76 meq/100 g of clay for the sample *Bofe*, to 104 meq/100 g of clay for the samples *Brasgel* and *Brasgel PA*. The surface area ranged from 593 m^2/g for the sample *Bofe* to 811 m^2/g for the samples *Brasgel* and *Brasgel PA* (Table 2); these values are typical for bentonites[24]. The industrialized samples presented larger values of CEC and specific area; this behavior is due to the fact that these clays are in the sodic form and are easily dispersed.

3.1.3. Chemical analysis

The loss on ignition (LI) represents the loss of inserted water, water from hydroxyl groups on the clay minerals, organic matter and carbonates[24]. The loss on ignition ranged from 8.59%, for the sample *Brasgel PA*, to 20.47%, for the sample *Chocolate* (Table 3). Fe_2O_3 ranged from 6.83%, for the sample *Bofe,* to 8.78%, for the sample *Verde-lodo* (Table 3). The amounts of Fe_2O_3, are derived from the crystalline lattice of illite, that contains about 4% to 6% of Fe_2O_3, and from the clay minerals of the smectite group, that is, montmorillonite or members from the series nontronite-beidellite, according to Souza Santos[24].

Table 3. Chemical composition of the natural and industrialized bentonites.

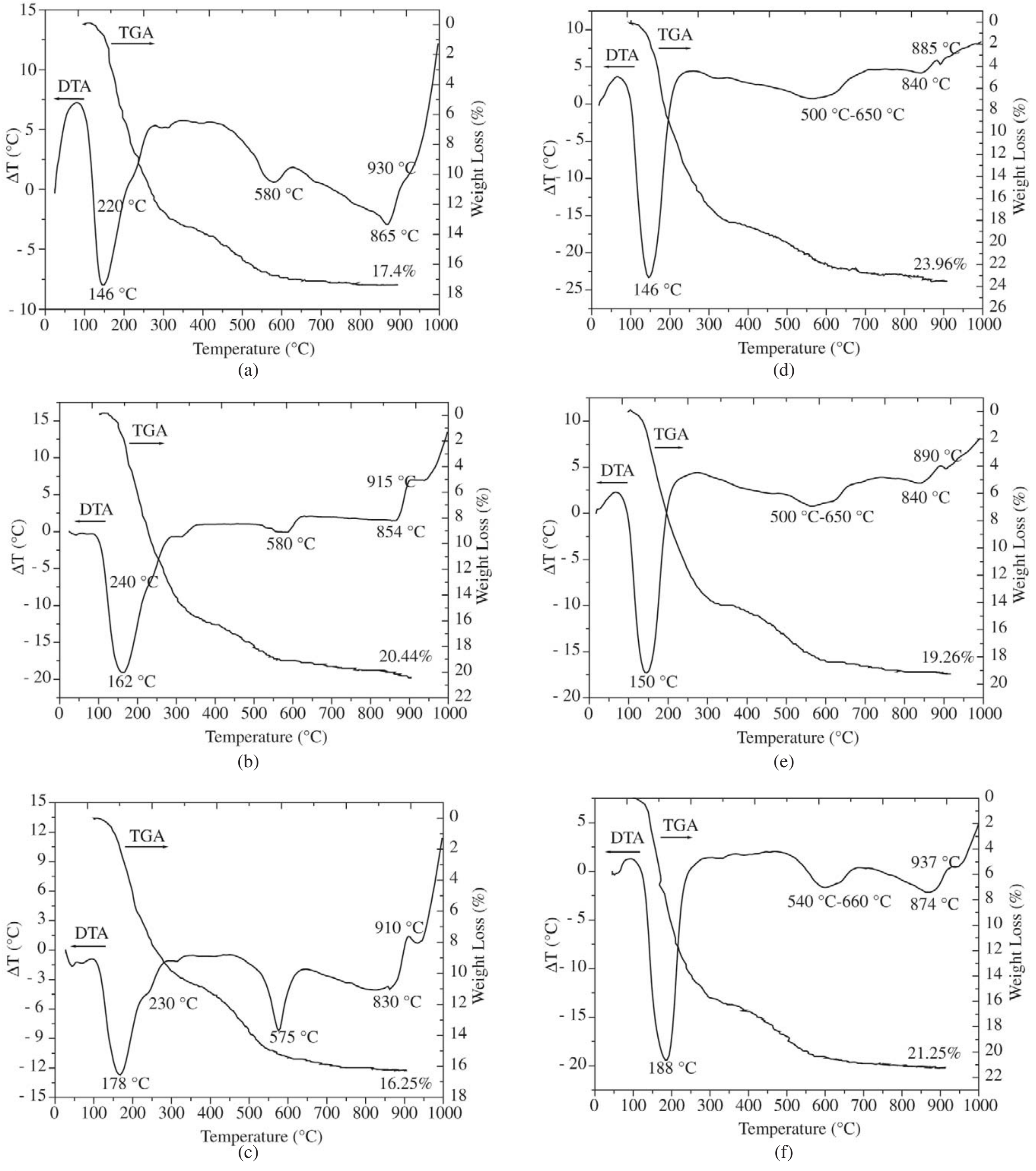
Samples	Chemical analyses (%)								
	LI*	IR**	SiO_2	Fe_2O_3	Al_2O_3	CaO	MgO	Na_2O	K_2O
Bofe	18.42	1.79	54.97	6.83	16.82	Trace	Trace	0.38	0.15
Chocolate	20.47	2.74	46.10	7.66	21.09	Trace	Trace	1.68	0.22
Verde-lodo	15.43	2.20	45.68	8.78	23.65	Trace	Trace	1.82	1.99
Dolomil	20.42	2.51	45.93	7.54	20.10	Trace	Trace	2.84	0.34
Brasgel	18.37	2.14	51.69	7.19	18.76	Trace	Trace	1.50	0.23
Brasgel PA	8.59	1.49	55.46	7.88	22.00	Trace	3.00	1.00	0.50
*Media****	16.30	ND	51.10	6.78	17.30	0.52	3.46	0.58	0.55

*LI - loss ignition at 1000 °C, **IR - insoluble residue, ***Sample studied by Souza Santos[6] when the deposit was discovered in Boa Vista, PB and ND – not determined.

All samples included traces of calcium oxide (CaO) and magnesium oxide (MgO). The MgO content of *Brasgel PA*, was 3.00%. The sample *Verde-lodo* contained 1.99% of K_2O, which is more than the other samples.

3.1.3. Thermal analysis

The differential thermal analysis and thermogravimetric curves of the clays are presented in Fig. 2. They are similar and typical for bentonites. The differential thermal curves



(a) (d)

(b) (e)

(c) (f)

Figure 2. Differential thermal analysis and thermogravimetric curves for the bentonites: a) *Bofe*; b) *Chocolate*; c) *Verde-lodo*; d) *Dolomil*; e) *Brasgel;* f) *Brasgel PA*.

present endothermic peaks characteristic of adsorbed water and hydroxyl groups from the clay mineral structure. Also the curve presents endo-exothermic peaks characteristic from formation of α or β-quartz of mullite. The thermogravimetric curve presented a slope related to the loss of hygroscopic water and hydroxyl groups.

The samples *Bofe*, *Chocolate* and *Verde-lodo* present undulations at approximately 220 °C, 240 °C and 230 °C, respectively. These are due to the presence of water coordinated to calcium and magnesium. This confirms the nature of polycationic clays. It was also observed that all the samples presented an endothermic band between 500 °C and 650 °C, characteristic of the hydroxyl loss of bentonite rich in iron.

3.1.4. X-ray diffraction

The X-ray diffraction pattern of the samples (Fig. 3) presents typical diffractograms of bentonite, with a presence of clay minerals from the smectite group. These are confirmed by the change of its characteristic peak, after treatment with ethylene glycol, from 14-15 Å to 17 Å. All the samples presented a peak at 3.35 Å, characteristic of the presence of the quartz. For the sample *Verde-lodo* (Fig. 3) a peak at 7.19 Å was observed, characteristic of kaolinite and a band between 4.55 Å and 4.35 Å, with superposition of peaks, characteristic of kaolinite, illite and clay minerals from the smectite group, probably montmorillonite or member from the nontronite-beidellite series.

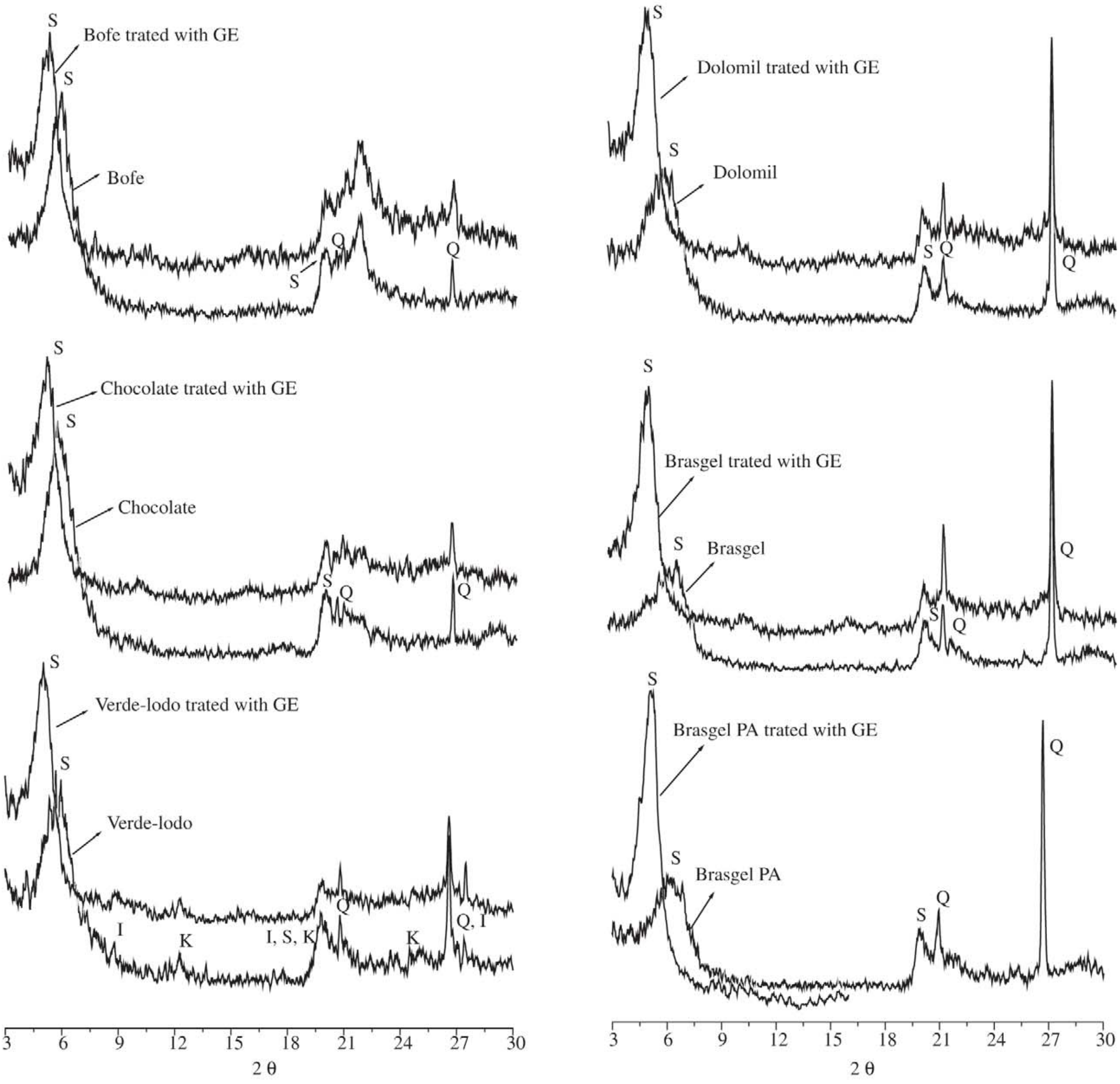


Figure 3. X-ray diffractions patterns for the bentonite samples: a) *Bofe*; b) *Chocolate*; c) *Verde-lodo*; d) *Dolomil*; e) *Brasgel;* f) *Brasgel PA*.

3.1.5. Infrared spectroscopy

The infrared spectrums of the clay samples are presented in Fig. 4. The spectra of the samples are quite similar, with Si-O-Si stretching vibration band that manifests at 1039.8 cm^{-1}, O-H stretching bands situated at 3626.25 cm^{-1} and 3425.08 cm^{-1} and a stretching and bending vibration of hydration water that are manifested at 1635 cm^{-1}. The set of the weak bands, which occurred in the range 920-550 cm^{-1} is ascribed to R-OH vibrations. According to Mendioroz *et al.*[25], the octahedral layers occurred at 920, 800 and 525 cm^{-1}. According to Srasra *et al.*[26], the tetrahedral bending modes of Al-O-Si, Si-O, Si-O-Mg and Si-O-Si of the clay minerals occurred at 545, 472 and 429 cm^{-1}, respectively.

3.1.6. Transmission electron microscopy

The micrographs of the samples (Fig. 5) presented typi-

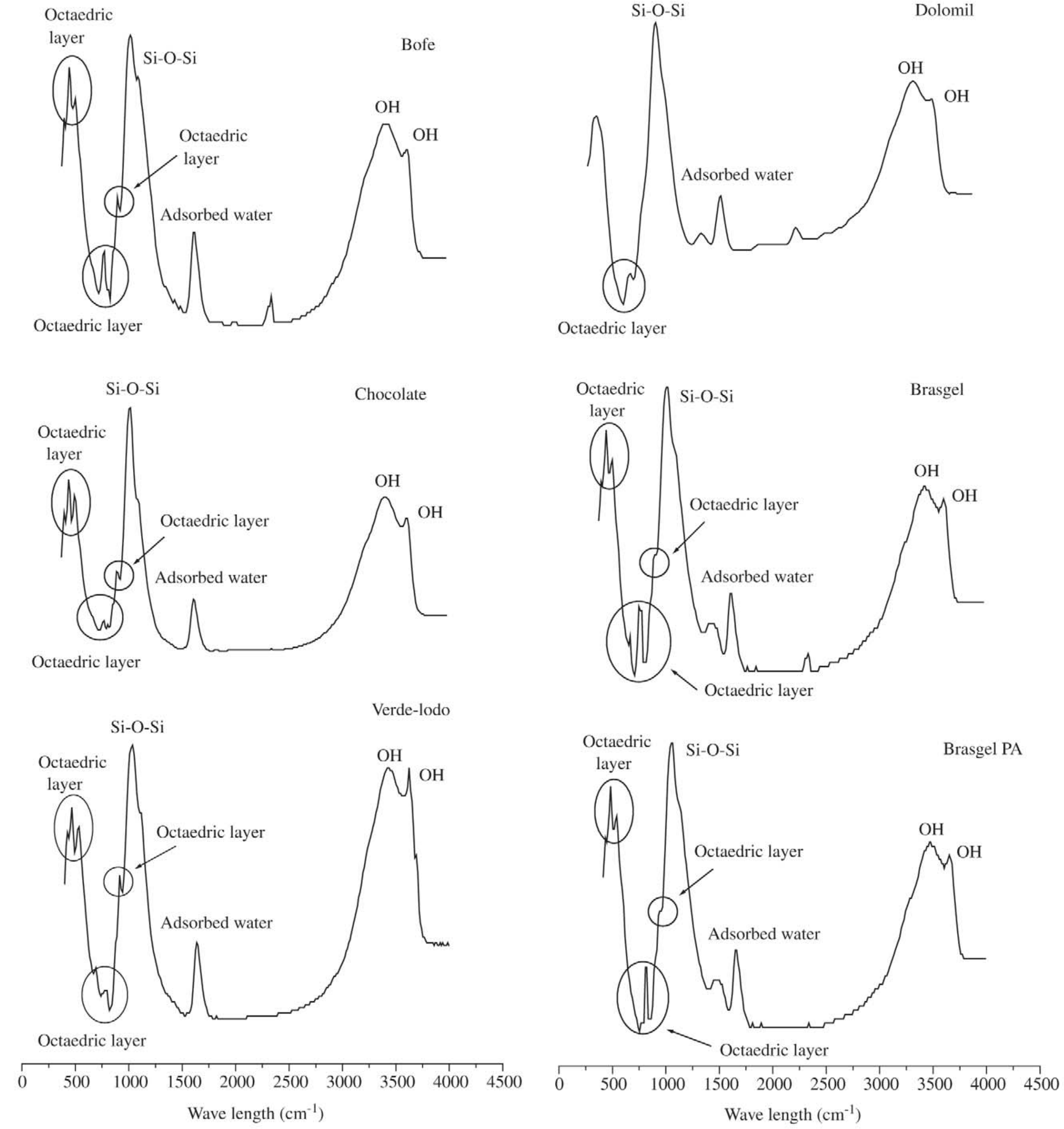


Figure 4. Infrared spectrums for the bentonite samples: a) *Bofe*; b) *Chocolate*; c) *Verde-lodo*; d) *Dolomil*; e) *Brasgel;* f) *Brasgel PA*.

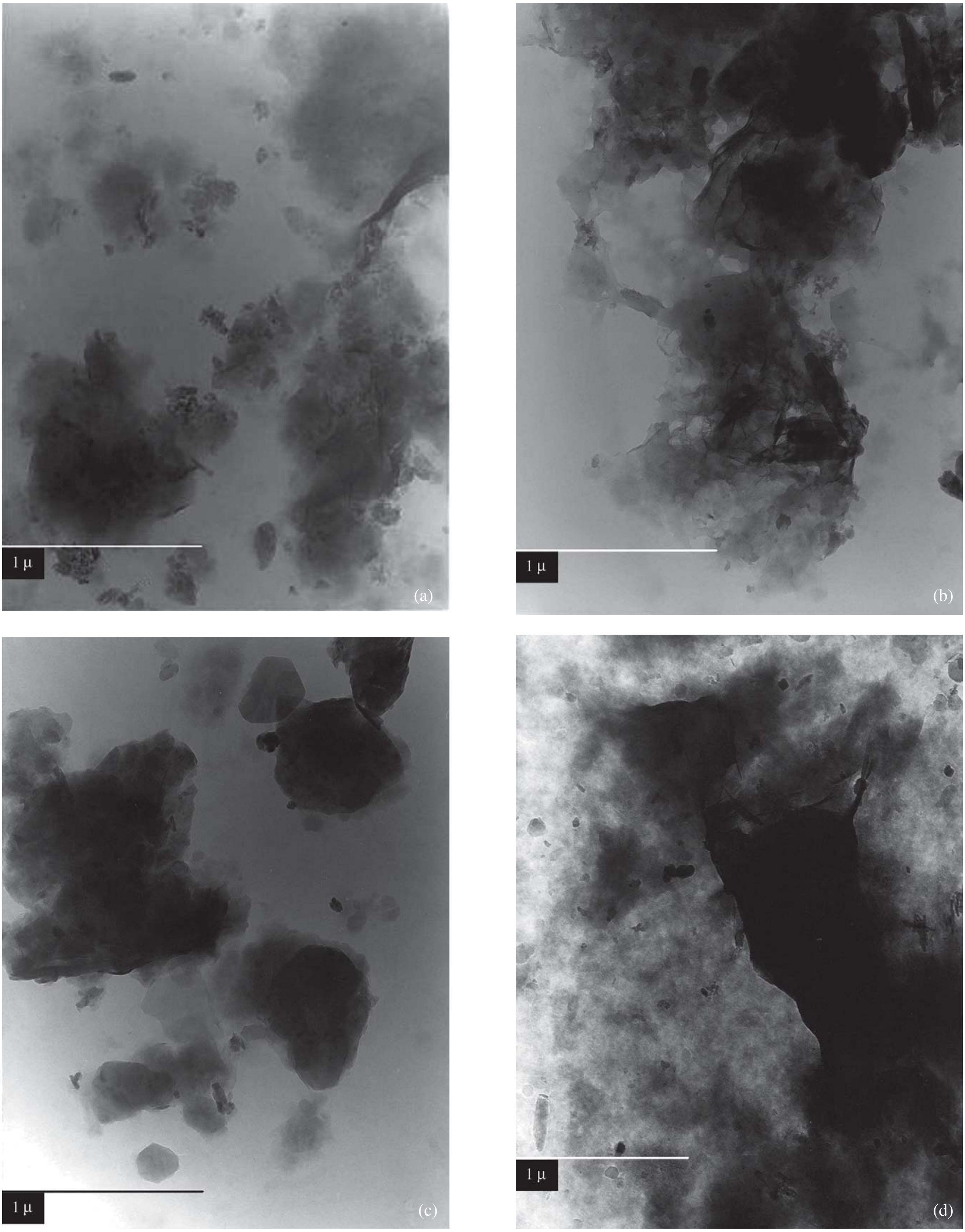


Figure 5. Micrographs of the bentonite samples: a) *Bofe*; b) *Chocolate*; c) *Verde-lodo*; d) *Dolomil*; e) *Brasgel;* f) *Brasgel PA*. Continue next page.

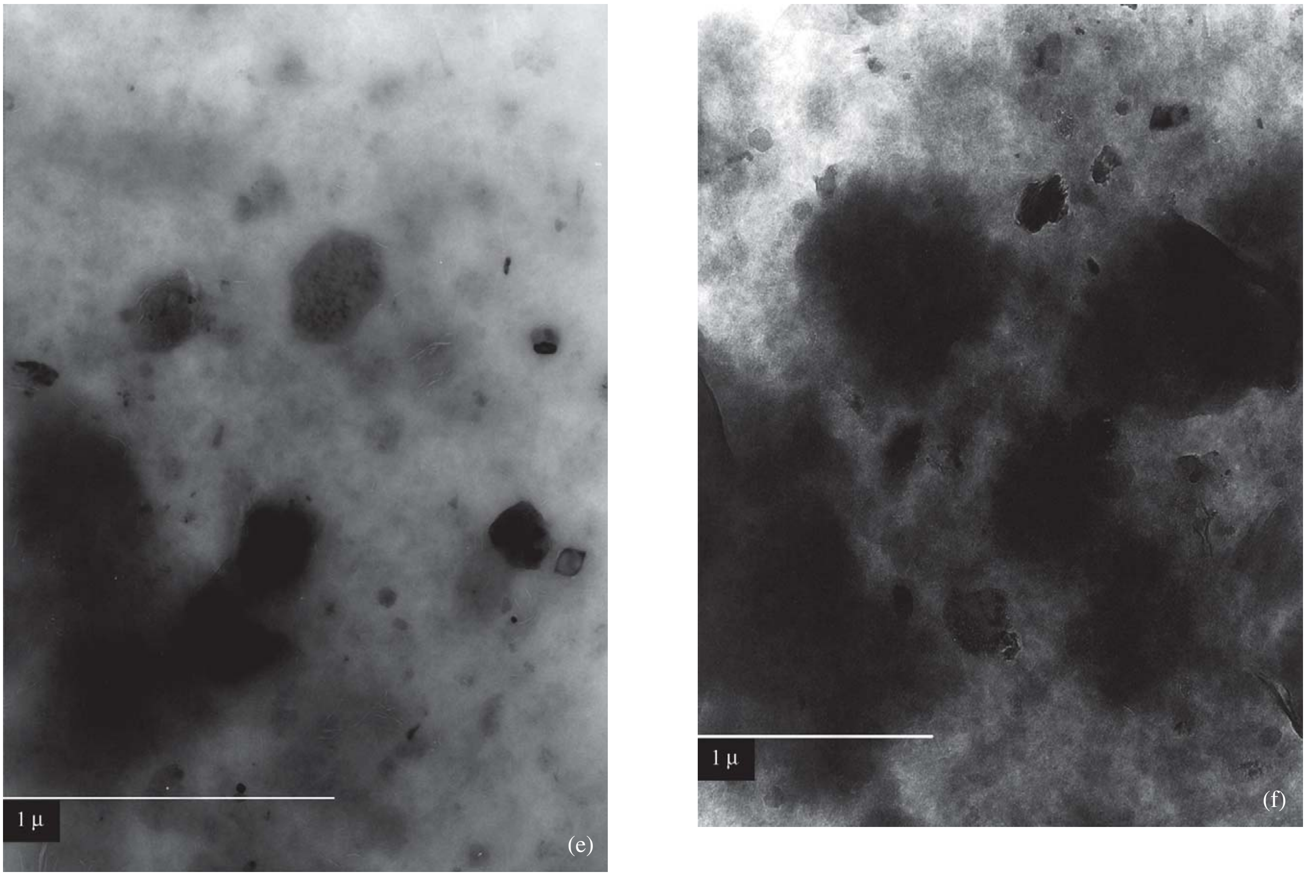


Figure 5. Micrographs of the bentonite samples: a) *Bofe*; b) *Chocolate*; c) *Verde-lodo*; d) *Dolomil*; e) *Brasgel;* f) *Brasgel PA*.

cal aspects of clay minerals from the smectite group, with irregular profile particles, undefined shape and great tendency to present curled edges, probably due to small plate diameter. Also, some irregular flake-shaped aggregates can be seen due to the attraction between the particles. In the micrograph (Fig. 5c) for sample *Verde-lodo* particles with hexagonal profile and lath-shapes can be observed, indicating the presence of kaolinite and illite, respectively, confirmed by X-ray results.

3.2. Rheological behavior

The rheological properties of the bentonite suspensions are given in Table 4. The increase in the proportion of Na_2CO_3 provide to the dispersions prepared with the natural clays an increase on apparent viscosity (AV) and decrease on plastic viscosity (PV) and water loss (WL), except for the sample *Bofe*, where PV did not present significant variations. This behavior shows the capacity of the natural clays to transform to the sodic form. The amount of Na_2CO_3 that gave best results was: 150 meq/100 g of dry clay, for the sample *Bofe*; 75 meq/100 g of dry clay, for the sample *Chocolate*; and 100 meq/100 g of dry clay, for the sample *Verde-lodo*.

Comparing the results presented in the Table 4 with the Petrobras specifications[20], it was observed that: i) the dispersions with *Bofe* presented values of AV lower than the minimum (15 cP). For the *Chocolate* and *Verde-lodo* samples AV were greater than 15 cP, except for *Chocolate* treated with 50 meq/100 g of dry clay and *Verde-lodo* treated with 50 and 75 meq/100 g of dry clay; ii) for PV, only the dispersions with *Bofe* clay, treated with all different proportions of Na_2CO_3, and *Verde-lodo*, treated with 50 meq/100 g of dry clay, presented values in agreement with the specifications, however very close to the minimum value (4.0 cP); and iii) among the studied clays, only *Chocolate*, treated with all proportions of Na_2CO_3, and *Bofe*, treated with 150 and 175 meq/100 g of dry clay, presented values of WL below the maximum (18.0 mL), according to Petrobras specifications[20]. The pH values were close to the maximum value (10.0).

For industrialized clays the apparent viscosity changed from 8.3 cP for the sample *Brasgel* to 16.1 cP for the sample *Brasgel PA*. For the plastic viscosity the values change from 4.0 cP, for *Brasgel PA* to 5.0 cP, for *Brasgel*. The water loss changes from 20.5 mL, for the samples *Dolomil* and *Brasgel*, to 16.3 mL, for the sample *Brasgel PA*. The

Table 4. Rheological properties of the suspensions prepared with the natural clays activated with Na_2CO_3 and of the suspensions prepared with the industrialized clays.

Natural samples	Amount of Na_2CO_3 (meq/100g of dry clay)	Properties rheological			pH
		AV (cP)	PV (cP)	WL (mL)	
Bofe	75	7.1	4.3	18.8	9.9
	100	8.1	4.5	18.5	10.0
	125	10.0	4.0	18.5	10.0
	150	12.5	4.8	17.7	10.0
	175	8.3	4.3	17.8	10.3
Chocolate	50	13.6	3.8	16.5	9.5
	75	18.5	3.8	16.0	9.9
	100	19.6	3.0	15.3	10.0
	125	18.8	2.3	15.8	10.0
	150	21.5	1.3	16.5	10.0
Verde-lodo	50	9.5	4.0	21.8	10.0
	75	12.9	2.0	22.5	10.1
	100	16.9	1.5	22.0	10.2
	125	18.9	1.3	23.5	10.2
	150	20.4	1.0	23.8	10.0
Dolomil		11.8	4.3	20.5	9.9
Brasgel		8.3	5.0	20.5	9.8
Brasgel PA		16.1	4.0	16.3	9.9
Standard values[23]		≥ 15.0	≥ 4.0	≤ 18.0	≤ 10.0

values of pH were close to 9.8, for all the samples. Comparing these results with the specifications[20] for water based drilling fluids, it was observed that the values of AV presented by the suspensions prepared with the industrialized clays were inferior to the Petrobras specifications (15 cP)[20], except for the suspensions prepared with the sample *Brasgel PA* that presented AV equal to 16,1 cP. The values obtained to PV, are in agreement with the specifications, however very close of the minimum value (4.0 cP); except for the sample *Brasgel* (5.0 cP). For WL, only the suspensions prepared with the sample *Brasgel PA* presented values below the maximum (18.0 mL). In relation to the pH values, all the samples presented results close to the maximum value (10.0).

4. Discussion

The water content of the samples is very low when compared with the Petrobras specifications (maximum 14%)[20] and can cause hydration problems if the clays is dry in high temperatures (above 60 °C). According to Souza Santos[24], the hydration of the bentonite could be related with the presence of the potassium in its structure. During the dry process, when the interlayer water is excluded, the clays containing potassium can acquire a crystalline structure similar to muscovite mica. This modification hinders the water penetration between the layers. Consequent suspension of the clay in water causes small values of apparent and plastic viscosities. Among the studied clays, only the *Verde-lodo* sample presented a high content of K_2O (1.99%), which can affect the rheologic properties. The amount of K_2O in this sample is several times superior than when it was discovery, according to the Souza Santos[6] results. The calcium, magnesium and sodium oxides of the natural clays also differ from the results presented by Souza Santos 30 years ago, and are inferior when compared with today results. The amounts of Na_2O and loss on ignition were superior. Also it is important to say the amount MgO decrease from 3.5% to traces.

The thermodifferential curves and X-ray diffraction of the natural (*Bofe*, *Verde-lodo* and *Chocolate*) and industrialized clays confirm that these sample are predominantly composed of clay minerals of the group of the smectite, probably montmorillonites or members of the series nontronite-beidellite. All the samples contained quartz in its composition. The sample *Verde-lodo* contained illite and kaolinite. The presence of these clay minerals can affect the process of sodium transformation of bentonites, and consequently influence its rheological behavior. The presence of clay minerals montmorillonites in all the samples and illite and kaolinite in the sample *Verde-lodo* was confirmed by the electron micrographs. The mineralogical composition of the studied clays is similar to the clays studied by Souza Santos[6] 30 years ago.

The natural clays, after treated with a Na_2CO_3 solution, can be transformed into sodium form, with rheological properties that do not satisfy the specifications of Petrobras[20], to be used as water based drilling fluids. The reasons for this are probably due to the presence of calcium and magnesium, as bicarbonates, in the suspensions. The calcium and magnesium bicarbonates present high solubility and, when in solution, they are dissociated liberating the cations calcium and magnesium and can occupy the sodium positions in the structure of the clay, with reversible cation exchange. Although the mineralogical compositions of the clays are similar to the ones tested, the rheological properties showed that the clays do not present the same values of apparent and plastic viscosities. In some samples the presence of other minerals and/or non-montmorillonite clay minerals, probably contributed to its behavior with flocculated structure and gel formation of the suspensions.

Among the industrialized clays, only the sample *Brasgel PA* presented rheological properties in agreement with the Petrobras specifications[20].

5. Conclusions

The study of the physical, mineralogical and rheological properties of three samples of clays natural bentonites and three industrialized ones from Boa Vista, Brazil, led to the following conclusions:

- the physical properties presented by the samples are in agreement with the specifications of Petrobras[20];
- the clay samples are composed of clay minerals from the smectite group, probably, montmorillonite or members of the series nontronite-beidellite. Also other minerals such as quartz and other clay minerals such as kaolinite and illite are included in the sample *Verde-lodo*;
- the natural clays, when treated with Na_2CO_3 solution can be transformed into sodium form. However the rheological properties do not satisfy the *Petrobras* specifications;
- among the industrialized clays, only *Brasgel PA* presented rheological properties fit to be used as a component of water based drilling fluids;
- after four decades of exploitation, the bentonite from Boa Vista presents mineralogical composition similar to the one from the beginning of industrialization but its rheological properties became so different and the results show a drop in the quality of these clays.

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