



Agronomía Colombiana

ISSN: 0120-9965

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Colombia

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four dispersant solutions  
Agronomía Colombiana, vol. 33, núm. 2, 2015, pp. 253-260  
Universidad Nacional de Colombia  
Bogotá, Colombia

Available in: <http://www.redalyc.org/articulo.oa?id=180341167015>

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# Methods of mechanical dispersion for determining granulometric fractions in soils using four dispersant solutions

## Métodos de dispersión mecánica en la determinación de fracciones granulométricas del suelo utilizando cuatro soluciones dispersantes

Katerine Borja<sup>1</sup>, Jaime Mercado<sup>1</sup>, and Enrique Combatt<sup>1</sup>

### ABSTRACT

Sieve analysis studies depend on obtaining suspensions of fully-dispersed and stable samples to facilitate the quantification of the fractions of soil aggregates. The aim of this study was to compare the percentage of fractions obtained with four chemical dispersants and two methods of mechanical dispersion. To carry out this investigation, nine soils were selected from the departments of Cordoba and Sucre and four methodologies using chemical dispersions:  $((\text{NaPO}_3)_6 + \text{Na}_2\text{CO}_3)$ ,  $(\text{NaOH})$  1M,  $(\text{Na}_4\text{P}_2\text{O}_7)$  0.1M pH 10, and  $\text{CH}_3\text{COONa}$  1M, and two methodologies of mechanical dispersion were evaluated: a slow one at 60 rpm for 6 hours and another at 4000 rpm for 15 minutes. The results were analyzed using a correlation test and contrasts. It was verified that the highest content of clay in the soil samples was found when using the 60 rpm agitation methodology, due to greater dispersion of the granulometric fractions. Likewise, when comparing the different methods of chemical dispersion, it was determined that NaOH had the highest dispersing ability and sodium acetate presented a low efficiency in the separation of soil particles.

**Key words:** granulometric analysis, clay, texture, mechanical agitation.

### RESUMEN

El estudio de un análisis granulométrico depende de la obtención de suspensiones de suelo completamente dispersas y estables para facilitar la cuantificación de las fracciones de los agregados del suelo. El objetivo de este trabajo fue comparar el porcentaje de fracciones obtenido con cuatro dispersantes químicos y dos métodos de dispersión mecánica. Para la realización de esta investigación fueron seleccionados nueve suelos de los departamentos de Córdoba y Sucre, en ellos se evaluaron cuatro metodologías de dispersión química:  $((\text{NaPO}_3)_6 + \text{Na}_2\text{CO}_3)$ ,  $(\text{NaOH})$  1M,  $(\text{Na}_4\text{P}_2\text{O}_7)$  0,1 M a pH 10,  $\text{CH}_3\text{COONa}$  1M y dos metodologías de dispersión mecánica, una lenta a 60 rpm durante 6 horas y la otra a 4000 rpm durante 15 minutos. Los resultados obtenidos fueron analizados mediante una prueba de correlación y contrastes. Se encontró que la agitación a 60 rpm durante 6 horas genera una mayor dispersión del suelo, al ser comparada con la agitación a 4000 rpm durante 15 minutos y el mayor contenidos de arcilla en las muestras de suelo se encontró cuando se utiliza agitación a 60 rpm. De igual forma al comparar los diferentes métodos químicos de dispersión, se determinó que el NaOH tiene la mayor capacidad dispersante y el acetato de sodio presenta poca eficiencia en la separación de fracciones granulométricas.

**Palabras clave:** análisis granulométrico, arcilla, textura, agitación mecánica.

## Introduction

Soil texture is an important physical property that helps determine appropriate soil management for increasing soil porosity and water infiltration. Silva *et al.* (2008) indicated that soil particle size has been used as an independent variable in pedotransfer functions to estimate more complex physical properties. Santos *et al.* (2010a) expounded that the determination of granulometric fractions allows the use of more rational and efficient fertilizers. According to Silva *et al.* (2004), the granulometric quantification of soils is obtained with textural analyses, which contribute to the description, classification and identification of soils.

The agricultural soils of the department of Cordoba, which may seem similar in their physical properties, may have differences; because of this, they tend to have different properties and performances when they are fertilized and mechanized. This is explained by their intrinsic characteristics and by the heterogeneity that exists in their physical properties; among these, soil texture has great relevance to appropriate agronomic management plans. Determining clay, sand and silt contents of soils is of great importance for agricultural production because, based on the percentage of each of these fractions, the appropriate management of irrigation, fertilization and acidity correction practices, among others, can be applied.

Received for publication: 14 November, 2014. Accepted for publication: 30 June, 2015.

Doi: 10.15446/agron.colomb.v33n2.47236

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The study of granulometric fractions carried out in laboratories is based on the velocity of particle sedimentation in a suspension or a solution (soils, dispersant and water) and after chemical and physical dispersants have been applied. Rodrigues (2008) stated that, in various experiments, these methodologies have been carried out, seeking to establish appropriate methods and procedures, but only a few have considered dispersion mechanisms and the diverse types of clay minerals present in each soil type. Ferreira (1999) explained that the complete dispersion of a soil sample is obtained with the integration of physical and chemical methods.

Concerning chemical dispersion, the most used agents are composed of a sodium salt, such as hexametaphosphate and sodium hydroxide, but, without question, the selection of the dispersant will depend on the cations that are present in the exchange complex and on the relation between the permanent and dependent charges of the pH which are found in the soil colloids. Rodrigues *et al.* (2011) found that the application of NaOH was efficient in many soils due to the increase of pH, by generating negative charges on the surface of the colloids and displacing cations with the Na in Oxisols soils. Corá *et al.* (2009) stated that the size and stability of aggregates increase with concentrations of humic and fulvic acids and, in tandem, with increases of oxides and hydroxides, Fe and Al, respectively; therefore, the use of a dispersant such as sodium hexametaphosphate is recommended because Na diminishes the osmotic pressure of the suspension, which contributes to the dispersion of the aggregates present in the soil.

On the other hand, mechanical dispersion procedures have been used at different levels of agitation with the objective of finding the greatest percentage of dispersion of the aggregates. To obtain these results, propeller type shakers have been used in the routine methodologies of soil laboratories because of the ease and speed of the processes. Oliveira *et al.* (2002), when comparing various methods of mechanical dispersion associated with chemical treatments, concluded that the process of slow agitation, with a horizontal helicoidal movement for three hours with an addition of 30 g of thick, coarse sand as an abrasive, was more efficient than the fast agitation method, with electrical mixer type equipment for the dispersion of Oxisol particles that are rich in gibbsite. Dourado *et al.* (2012) found satisfactory efficiency when using a slow reciprocating shaker for mechanical dispersion of soil samples of different texture classes. Mechanical analysis, even though being a traditional method used in soil sample laboratories, still presents a significant disparity in its results, mainly due to the high heterogeneity

among the different analytical methodologies employed (Rodrigues *et al.*, 2011).

In Colombia, soil sample laboratories determine granulometric fractions using the same analytic methodologies without bearing in mind factors that gain influence during quantification, such as the different sizes of particles and aggregation which are conditioned by the presence of elements such as Ca, Al, Fe, Mn and different organic compounds generated from the synthesis of microorganisms. The purpose of this study was to compare the dispersion capacity of two methods of mechanical agitation, using different chemical solutions as dispersing agents.

## Materials and methods

For this experiment, nine soil samples of approximately 10 kg were collected from several municipalities in the departments of Córdoba and Sucre. Sub-samples of 1 kg from each soil sample were processed at the Soil and Water Laboratory of the Faculty of Agricultural Sciences of the Universidad de Córdoba, according to the norms established by the IGAC (2006) (*Instituto Geográfico Agustín Codazzi*) for their chemical characterization with the following parameters: pH; organic matter by the Walkley-Black test;  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$ ,  $\text{Na}^+$ ,  $\text{K}^+$  extracted with 1M ammonium acetate pH 7 and quantified by spectrophotometric absorption and by atomic emission, respectively, with a Perkins-Elmer 3110 spectrophotometer (Norwalk, CT); exchangeable acidity extracted with KCl 1 M. In addition, the samples were subjected to mineralogical analysis for clay and sand (IGAC).

To carry out the soil texture analysis, 60 g of each soil sample were placed in plastic containers; the soils were dried, ground and screened at 2 mm. To this quantity of soil, 10 mL of solution, depending on the type of chemical dispersant used, were added. The chemical dispersion of the samples was done using four dispersing agents: Calgon ( $(\text{NaPO}_3)_6 + \text{Na}_2\text{CO}_3$ ), sodium hydroxide (NaOH) 1M, sodium pyrophosphate ( $\text{Na}_4\text{P}_2\text{O}_7$ ) 0.1 M at pH 10, and sodium acetate ( $\text{CH}_3\text{COONa}$ ) 1 M. In addition to the chemical dispersants, 200 mL of water were added to each of the treatments.

For the mechanical dispersion using a slow agitation process at 60 rpm, the soils, dispersants and water were placed in a polypropylene container, adding approximately 200 mL of water. Then, the containers with soils and dispersants (treatments) were allowed to settle for 24 h and, after this time, each treatment was subjected to agitation, using an agitator for a period of 6 h.

The fast mechanical dispersion was carried out with a Jika-trademarked, propeller-equipped device for a period of 15 min. at 4000 rpm. The suspension was placed in a polypropylene container adding 200 mL of water. After this time, each soil was subjected to agitation and, after this process, the soil samples were allowed to settle for 24 h.

For the quantification of the granulometric fractions obtained after the agitation processes (slow and fast), the solutions were transferred to a sedimentation cylinder and water was added to bring the volume to 1000 mL with the hydrometer inside. Following this, the suspensions were manually agitated for approximately 2 min and the hydrometer was read at 40 s and 2 h after the agitation process, reading the temperatures at both times to obtain the correction factor. With this procedure, it was possible to determine the percentages of clay and sand in each of the treatments using chemical and mechanical dispersants whit three repetitions per treatment.

To compare the dispersion capacity of the chemical agents within each of the mechanical agitation procedures, the procedure proposed by Leite and Oliveira (2002) was used. Contrasts and comparisons between the tested dispersants were also carried out ( $P \leq 0.05$ ).

The Leite and Oliveira (L&O) test consisted of a decision rule built upon an F statistics basis as proposed by Graybill (1976) in the evaluation of the medium error in the analysis of the coefficients of the lineal correlation. According to the authors,  $Y_j$  and  $Y_i$  are two quantitative vectors, in which  $j$  indicates an alternative method, procedure or treatment and  $i$  is the treatment standard.

According to the authors, the two methods are considered statically equal if, simultaneously after the lineal regression adjustment,  $Y_i = \beta_0 + \beta_1 Y_j$ ,  $\beta_0 = 0$  and  $\beta_1 = 1$ , with the  $R_{Y_j Y_i}$  value very close to 1. Because this situation does not really occur, the authors carried out a more exhaustive statistical analysis, in which tests of  $t$  and  $F$  were done simultaneously, evaluating their significance; furthermore, a medium error test and a correlation analysis were carried out, where  $R$  should have been close to one. When evaluating all these factors, the following rules of test decision were present in Tab. 1.

## Results

In the characterization analysis carried out (Tab. 2), it can be observed that soils with different pH levels were used, which varied between 4.7 and 8.0; including the following

**TABLE 1.** Decision rules of the Leite and Oliveira test for the statistical analysis of the two compared methods for determining granulometric fractions in soils.

Situation	F( $H_0$ )	$t_{(e)}$	$R_{Y_j Y_i}$	Decision
1	NS	NS	$R_{Y_j Y_i} \geq (1 -  e )$	$Y_j = Y_i$
2	NS	NS	$R_{Y_j Y_i} \leq (1 -  e )$	$Y_j \neq Y_i$
3	NS	*	$R_{Y_j Y_i} \geq (1 -  e )$	$Y_j \neq Y_i$
4	NS	*	$R_{Y_j Y_i} \leq (1 -  e )$	$Y_j \neq Y_i$
5	*	NS	$R_{Y_j Y_i} \geq (1 -  e )$	$Y_j \neq Y_i$
6	*	NS	$R_{Y_j Y_i} \leq (1 -  e )$	$Y_j \neq Y_i$
7	*	*	$R_{Y_j Y_i} \geq (1 -  e )$	$Y_j \neq Y_i$
8	*	*	$R_{Y_j Y_i} \leq (1 -  e )$	$Y_j \neq Y_i$

ns: not significant F and t tests; \*: significant at  $P \leq 0.05$  and  $|e|$ : average error.

variations: organic matter of 0.5 to 2.9%; calcium between 0.4 and 87.0  $\text{cmol}^+ \text{kg}^{-1}$ ; magnesium between 0.1 and 10.0  $\text{cmol}^+ \text{kg}^{-1}$  and an exchangeable acidity of 0.4 to 6.2  $\text{cmol}^+ \text{kg}^{-1}$ .

When carrying out the mineralogical analysis of the clays, one can observe, in the same table, that the predominant minerals in the clay fraction were kaolinite and quartz. This demonstrates the high acidification and state of edaphological development of the soils, due to the intemperization that occurred in the rocks or parent materials that gave rise to soils and where the samples were collected. The presence of kaolinite and the high quantity of quartz present in these soils, conferred physical and chemical properties that resulted in a low or nil expansibility and a low capacity of cationic exchange in some of the samples; furthermore, there was gibbsite in the other soils, which provided oxide properties. The presence of these minerals contributed to the fact that these soils presented dependent pH charges, elevated points of zero charges and a high degree of flocculation of clays, which may have reduced the effect of deflocculant ions. According Stumm and Wieland (1990), the occurrence of  $\text{pH}_{\text{pzc}}$  is fundamentally important to many processes occurring in the mineral-water interface, including adsorption, dissolution, precipitation, and colloid formation.

Upon observing the results obtained in the quantification of the clays, independent of the chemical dispersants, it can be indicated that the highest content of this fraction was found when using the mechanical agitation methodology at 60 rpm (Tab. 3).

This was due to the mechanical dispersion capacity, which was based on two principal factors: the elevated kinetic energy that the particles acquired and the greater contact time between the dispersant and the soil sample during the agitation process. Vitorino *et al.* (2007) explained that the granulometric analysis of soils requires sufficient energy to overcome the bonding forces of the aggregates.

**TABLE 2.** Chemical and mineralogy characterization of the soil samples collected in departments of Cordoba and Sucre (Colombia).

Soils	pH	MO	Ca	Mg	K	Na	Al+H	ECEC	Clay mineralogy		
	1:1	%	cmol kg <sup>-1</sup>						Mica	Kaolinite	Quartz
1	7.8	0.51	87.4	1.7	0.83	0.30		90.2	++	tr	++
2	4.8	0.66	0.4	0.1	0.09	0.11	6.20	6.9	+	+++	+
3	6.6	2.93	13.0	6.7	0.45	0.35		20.4	++	+++	++
4	4.7	0.98	1.5	0.8	0.21	0.11	0.40	3.0	tr	++	++++
5	5.1	1.15	0.5	0.8	0.13	0.09	0.62	2.1		++	++++
6	6.5	0.99	11.0	5.8	1.54	0.20		18.5	+++	++	+
7	5.0	2.77	10.5	10.0	0.51	0.33	0.70	22.0	++	++	+
8	7.5	1.20	45.0	10.0	0.28	0.43		55.7	+	tr	+
9	8.0	1.20	42.5	0.8	0.32	0.11		43.7	tr	+	+
Minimum	4.7	0.50	0.4	0.1	0.09	0.09	0.40	2.1	--	--	--
Maximum	8.0	2.93	87.4	10.0	1.54	0.43	6.20	90.2	--	--	--
Average	6.2	1.40	23.5	4.1	0.50	0.20	2.00	29.2	--	--	--
Standard deviation	1.3	0.90	29.3	4.1	0.50	0.10	2.80	29.1	--	--	--

Clay mineralogy: ++++ dominant (>50%); +++ abundant (30-50%); ++ common (15-30%); + present (5-15%); tr, traces (<5%).

**TABLE 3.** Descriptive statistics for the clay (%) determined through the use of different methods of chemical and physical dispersion for determining granulometric fractions in soils.

Soils	Agitation at 60 rpm for 6 h				Agitation at 4000 rpm for 15 min			
	Calgon	CH <sub>3</sub> COONa	NaOH	Na <sub>4</sub> P <sub>2</sub> O <sub>7</sub>	Calgon	CH <sub>3</sub> COONa	NaOH	Na <sub>4</sub> P <sub>2</sub> O <sub>7</sub>
1	37.45	2.69	38.40	39.36	29.67	2.05	30.14	30.62
2	51.26	8.40	61.26	50.79	38.50	6.60	57.55	42.31
3	33.40	6.42	38.88	35.39	30.10	2.56	38.52	29.55
4	15.79	8.64	18.17	16.74	14.14	6.52	17.48	13.19
5	22.45	10.55	24.83	23.40	17.00	7.95	22.24	18.43
6	35.31	2.93	36.74	35.79	32.69	2.21	31.74	27.93
7	49.76	11.67	52.62	50.71	43.95	16.33	45.38	42.52
8	53.10	14.05	50.71	47.86	35.55	16.50	45.07	40.31
9	48.33	3.10	46.43	45.95	34.60	2.69	35.07	37.93
Minimum	15.79	2.69	18.17	16.74	14.14	2.05	17.48	13.19
Maximum	53.10	14.05	61.26	50.79	43.95	16.50	57.55	42.52
Average	38.54	7.60	40.89	38.44	30.69	7.05	35.91	31.42
Standard deviation	13.28	4.13	13.61	12.05	9.63	5.75	12.38	10.49

It should be noted that the movement generated by the slow rotation of 360° resulted in a collision between the particles, which caused a separation of the aggregate particles.

Furthermore, with the six hours that this process required; the phenomenon of disintegration increased throughout the whole sample. Tavares-Filho and Magalhães (2008) explained that the slow agitation methodology generates a greater dispersion due to the homogeneity in the process, which obtains smaller-sized aggregates and, consequently, a greater clay content. Santos *et al.* (2010b) observed that slow agitation provides a greater physical dispersion compared with fast agitation, demonstrating a greater exactness and precision when observing the texture class of a soil.

When comparing the effects of mechanical agitation at 60 and 4000 rpm with the chemical dispersants, one can observe that the sand contents diminished when increasing

the pH of the dispersants. On average, in all of the evaluated soils with the dispersants, the sand percentage went from 35 to 30% for the agitation at 60 rpm and from 42 to 35% using agitation at 4000 rpm (Fig 1A and B).

In regards to the chemical dispersants, it was found that CH<sub>3</sub>COONa presented the lowest ability of granulometric fraction separation of the soils. This was due to the low sodium concentration present in the solution and the accompanying anion because this compound had acidic properties. A condition that was verified by quantifying the pH of the suspensions, which had an average of 6.1 for CH<sub>3</sub>COONa, ((NaPO<sub>3</sub>)<sub>6</sub> + Na<sub>2</sub>CO<sub>3</sub>), 8.42 for Na<sub>4</sub>P<sub>2</sub>O<sub>7</sub> and 8.47 for NaOH 11.51, independent of the agitation.

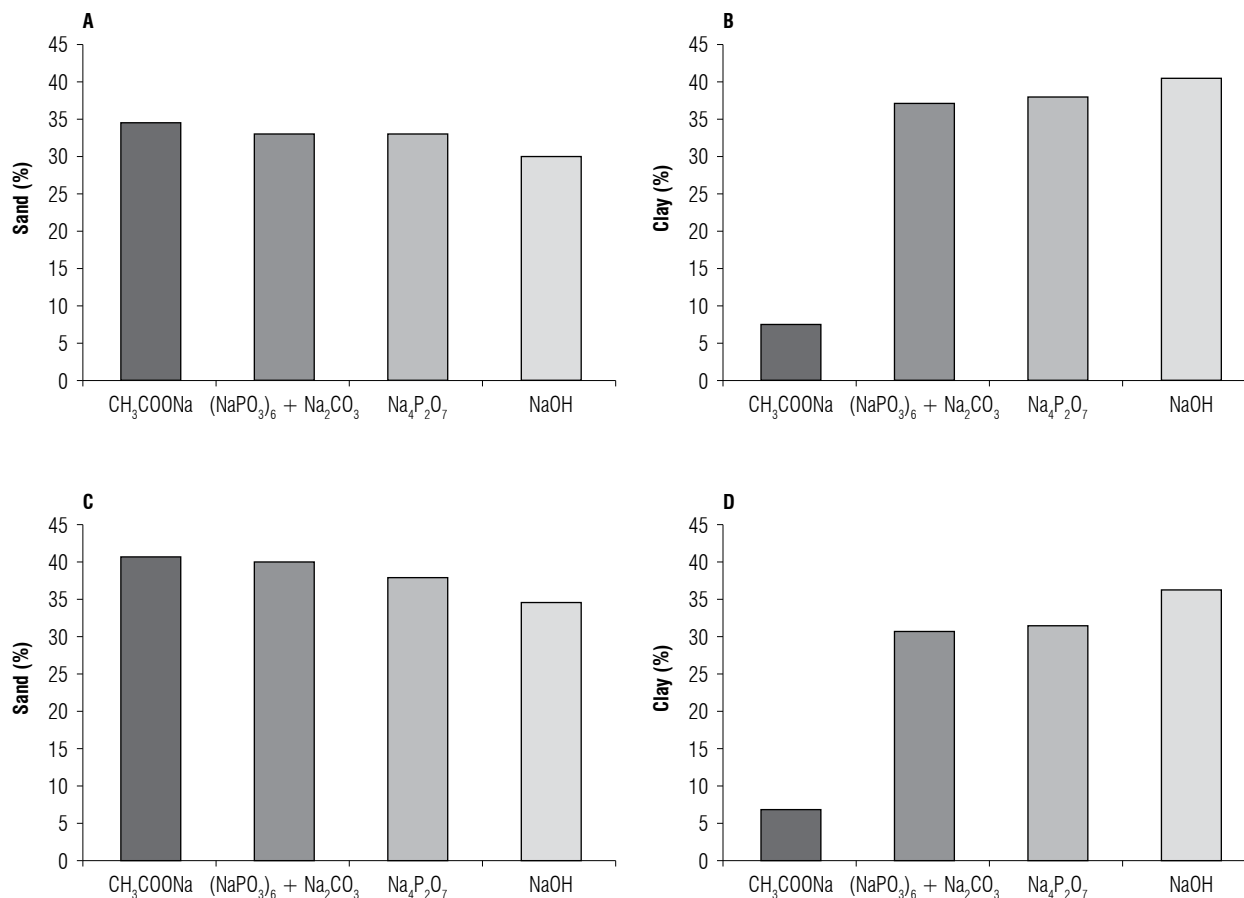
This increase in acidity countered the particle dispersion due to the electrostatic attraction that can be generated among and between aggregates; this promoted the

formation of flocculants. These results coincide with those reported by Medina and Grohmann (1962), who wrote that the stability in the solutions for the dispersion of the clays requires high pH levels to obtain an increase in the negative charges of the solution.

The maximum chemical dispersion grade was obtained using NaOH as a dispersant in the two mechanical methods, where the pH level in the solution was greater than 11.0. The dispersant effect of this chemical agent was due to a sodium concentration that permitted an increase in the expansion of the diffuse double layer, which in turn, allowed an increase in the dispersion of the granulometric fractions. According to Ruiz (2005), chemical dispersion is based on increasing the repulsion between particles and processes, which occurs by increasing the diffuse double layer by saturation of the complex cations exchange with Na. Furthermore, the chemical properties, such as the ionic radius, that Na has allow the ion to have a greater range of hydration, which contributes to the separation of particles in the soil. According to Spera *et al.* (2008), the thickness

of the diffuse double layer is altered by the concentration and type of electrolytes, mainly by ions such as Na and K.

The percentages of the evaluated clays indicated that there was a greater content of this fraction with the use of the NaOH dispersant, reaching values of 45 and 38% for agitation at 60 and 4,000 rpm, respectively (Fig. 1A and B). These results can be explained by the dispersant capacity of sodium present in this chemical solution, which contributed to the movement and exchange of the divalent and trivalent ions present in the diffuse double layer, which resulted in a greater repulsion among particles. These results coincide with those expressed by Tavares-Filho and Magalhães (2008), who concluded that the greatest dispersion occurred when using NaOH and slow agitation, but are different from those found by Jucksch (1995), who worked with concentrations of 0.01 M NaOH; he determined that the formation of floccules existed and explained that this was due to the concentration of NaOH, which caused a contraction of the diffused double layer and flocculation of the colloids.



**FIGURE 1.** Granulometric fraction quantities of the clay and sand as determined by chemical dispersants and agitation at 60 (A and B) and at 4,000 (C and D) rpm.

Concerning the capacity of the chemical dispersants (Fig. 1B and D), one can observe that NaOH was able to quantify a greater quantity of clay than the Calgon standard ((NaPO<sub>3</sub>)<sub>6</sub> + Na<sub>2</sub>CO<sub>3</sub>) used in Colombia. This efficiency can be attributed to the pH level that was in this solution, which had an approximate value of 11.0. Calvacante (2010) indicated that the optimum pH achieving the greatest level of dispersion occurs when solutions achieve a pH level near 12. Jucksch (1995) concluded that NaOH has a better dispersant action with an elevated pH function, up to a determined concentration where exchange reactions with absorbed cations exist. Mauri (2011), after evaluating different dispersion methods, stated that the greater the clay content extracted by chemical and physical dispersion methods, the better the method. Bearing this in mind and concerning the present study of different soils in Cordoba and Sucre, we can indicate that the method with the best dispersant capacity was 1.0 M NaOH.

When comparing the chemical dispersant Calgon with CH<sub>3</sub>COONa, it was found that the latter dispersant quantified the least percentage of clay, obtaining values that were quite inferior to the other dispersants. A low concentration of negative charges depends on pH, which is generated with CH<sub>3</sub>COONa, which in turn has an acidity that generates a reduction of the thickness of the diffused double layer, thereby causing a grouping of the particles and generating a flocculation phenomenon. Almeida Neto *et al.* (2009) explained that when the soil pH is close to the pH of the zero charge point, the positive and negative charges are equal; thus, the electrostatic repulsion powers are very weak, which tends to provide a reduced electric potential with many ions that remain in the diffused double layer.

The statistical analysis, by means of the Leite and Oliveira test, of the data that were obtained with the mechanical dispersion method that used agitation at 60 rpm and chemical dispersants to determine the sand fraction demonstrated that equality existed between the dispersants Calgon ((NaPO<sub>3</sub>)<sub>6</sub> + Na<sub>2</sub>CO<sub>3</sub>) and NaOH and with Na<sub>4</sub>P<sub>2</sub>O<sub>7</sub>, with correlation coefficients of 0.97 and 0.99, respectively. Spera *et al.* (2008) found that the thickness of the diffused double layer was altered by the concentration and types of electrolytes, mainly by ions such as Na and K. These results indicated that an elevated relationship exists between the studied methodologies; therefore, these chemical dispersants can be used for the quantification of the sand fraction with mechanical agitation at 60 rpm (Tab. 4).

The study of granulometric fractions with an agitation at 4000 rpm presented approximate correlations of 0.85 between the Calgon standard and the other methods used to determine the sand content. With this agitation method, there was an equality of methods between (NaPO<sub>3</sub>)<sub>6</sub> + Na<sub>2</sub>CO<sub>3</sub> and Na<sub>4</sub>P<sub>2</sub>O<sub>7</sub>.

These results indicate that, with an agitation of 60 and 4000 rpm and using the chemical dispersant Na<sub>4</sub>P<sub>2</sub>O<sub>7</sub>, similar amounts of sand can be found. Rodrigues (2008) found that the addition of PO<sub>4</sub> improves the dispersion of clay in Oxisols, Alfisols and Mollisols.

Upon determining the quantity of clay used in the agitation methodologies at 60 and 4000 rpm, no equality of methods was found between the standard procedure and the other methods of chemical dispersal. These results can be explained because significant differences were found for

**TABLE 4.** Identity test among the chemical dispersion methods in the quantification of the clay and sand fractions using agitation at 60 and 4,000 rpm, according to the Leite and Oliveira test.

Fractions	Y <sub>j</sub>	Y <sub>i</sub>	R <sup>2(1)</sup>	F(H <sub>0</sub> ) <sup>2</sup>	t <sub>(e)</sub> <sup>3</sup>	R <sub> Y<sub>j</sub>Y<sub>i</sub>  ≥ 1 -  e </sub>	Decision
<b>Agitation at 60 rpm</b>							
Sand	(NaPO <sub>3</sub> ) <sub>6</sub> + Na <sub>2</sub> CO <sub>3</sub>	CH <sub>3</sub> COONa	0.99	11.35*	3.35*	Si	Y <sub>j</sub> ≠ Y <sub>i</sub>
	(NaPO <sub>3</sub> ) <sub>6</sub> + Na <sub>2</sub> CO <sub>3</sub>	NaOH	0.97	3.15 NS	2.26 NS	Si	Y <sub>j</sub> = Y <sub>i</sub>
	(NaPO <sub>3</sub> ) <sub>6</sub> + Na <sub>2</sub> CO <sub>3</sub>	Na <sub>4</sub> P <sub>2</sub> O <sub>7</sub>	0.99	1.22 NS	1.53 NS	Si	Y <sub>j</sub> = Y <sub>i</sub>
Clay	(NaPO <sub>3</sub> ) <sub>6</sub> + Na <sub>2</sub> CO <sub>3</sub>	CH <sub>3</sub> COONa	0.02	21.9*	3.55*	Si	Y <sub>j</sub> ≠ Y <sub>i</sub>
	(NaPO <sub>3</sub> ) <sub>6</sub> + Na <sub>2</sub> CO <sub>3</sub>	NaOH	0.92	1.83 NS	2.44*	Si	Y <sub>j</sub> ≠ Y <sub>i</sub>
	(NaPO <sub>3</sub> ) <sub>6</sub> + Na <sub>2</sub> CO <sub>3</sub>	Na <sub>4</sub> P <sub>2</sub> O <sub>7</sub>	0.97	0.90 NS	0.37 NS	No	Y <sub>j</sub> = Y <sub>i</sub>
<b>Agitation at 4,000 rpm</b>							
Sand	(NaPO <sub>3</sub> ) <sub>6</sub> + Na <sub>2</sub> CO <sub>3</sub>	CH <sub>3</sub> COONa	0.85	0.79 NS	0.13 NS	No	Y <sub>j</sub> ≠ Y <sub>i</sub>
	(NaPO <sub>3</sub> ) <sub>6</sub> + Na <sub>2</sub> CO <sub>3</sub>	NaOH	0.83	4.89*	2.08 NS	Si	Y <sub>j</sub> ≠ Y <sub>i</sub>
	(NaPO <sub>3</sub> ) <sub>6</sub> + Na <sub>2</sub> CO <sub>3</sub>	Na <sub>4</sub> P <sub>2</sub> O <sub>7</sub>	0.86	1.18 NS	1.20 NS	Si	Y <sub>j</sub> = Y <sub>i</sub>
Clay	(NaPO <sub>3</sub> ) <sub>6</sub> + Na <sub>2</sub> CO <sub>3</sub>	CH <sub>3</sub> COONa	0.10	26.76*	3.50*	Si	Y <sub>j</sub> ≠ Y <sub>i</sub>
	(NaPO <sub>3</sub> ) <sub>6</sub> + Na <sub>2</sub> CO <sub>3</sub>	NaOH	0.74	6.99*	3.17*	No	Y <sub>j</sub> ≠ Y <sub>i</sub>
	(NaPO <sub>3</sub> ) <sub>6</sub> + Na <sub>2</sub> CO <sub>3</sub>	Na <sub>4</sub> P <sub>2</sub> O <sub>7</sub>	0.90	1.02 NS	0.38 NS	No	Y <sub>j</sub> ≠ Y <sub>i</sub>

ns: not significant F and t tests; \*:significant at  $P \leq 0.05$  and |e|: average error.

the T test, which was done in the statistics test, comparing  $(\text{NaPO}_3)_6 + \text{Na}_2\text{CO}_3$  with  $\text{CH}_3\text{COONa}$ ,  $(\text{NaPO}_3)_6 + \text{Na}_2\text{CO}_3$  and  $\text{NaOH}$  (Tab. 4).

The data on the amount of sand, clay and Silt were also analyzed statistically through contrast analysis (Tab. 5); the chemical dispersants were compared among the mechanical agitation methodologies. The statistical analysis indicated that significant differences did not exist between the quantities of the three granulometric fractions.

These results coincide with those of Ferreira (1999), who found that no statistically significant differences existed between the fast and slow agitation methods. But, the slow agitation method had a superior dispersant power. Sousa Neto *et al.* (2009), using  $\text{NaOH}$  and  $\text{Na}_{16}\text{P}_{14}\text{O}_3$  as dispersants and methods of fast and slow mechanical agitation, indicated that they did not find efficiency in the dispersion of clay with the use of these dispersants and that there were no differences among the mechanical agitations.

**TABLE 5.** Orthogonal contrast between the chemical dispersants with different agitation times.

Contrasts		Sand	Clay	Silt
60 rpm	4000 rpm		%	
$(\text{NaPO}_3)_6 + \text{Na}_2\text{CO}_3$	$(\text{NaPO}_3)_6 + \text{Na}_2\text{CO}_3$	-8.23 NS	7.85 NS	0.38 NS
$\text{CH}_3\text{COONa}$	$\text{CH}_3\text{COONa}$	-6.75 NS	0.56 NS	6.20 NS
$\text{NaOH}$	$\text{NaOH}$	-4.88 NS	4.99 NS	-0.10 NS
$\text{Na}_4\text{P}_2\text{O}_7$	$\text{Na}_4\text{P}_2\text{O}_7$	-5.35 NS	7.02 NS	-1.67 NS

NS, not significant.

Mauri *et al.* (2011), working with alternative dispersants, found that a mixture of  $((\text{NaPO}_3)_n + \text{NaOH})$  showed results close to the referenced dispersants, such as  $\text{NaOH}$ , evidence that stresses the importance of elevated pH values, up to or around 12.0.

## Conclusions

Sodium Hydroxide at 1.0 M is the most efficient dispersant for the quantification of tropical clay contents.

The increase of the pH level in the solution favored the dispersion of the granulometric fractions, increasing the clay content.

Sodium acetate is not efficient as a dispersant agent because it is not efficient in the separation of granulometric fractions.

Agitation at 60 rpm for 6 h generated a greater dispersion of the soils when compared with agitation at 4000 rpm for

15 min  $(\text{NaPO}_3)_6 + \text{Na}_2\text{CO}_3$  and  $\text{Na}_4\text{P}_2\text{O}_7$  presented a similar behavior for their dispersant capacities.

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