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Chemical dispersants and quantification textural fractions by Bouyoucos and pipette methods

Dispersantes químicos y cuantificación de fracciones texturales por los métodos Bouyoucos y pipeta

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Abstract

An adequate individualization of particles is required for a correct granulometric analysis in order to quantify the real percent composition of particles in the soil. The objective of this study was to compare the percent of granulometric fractions determined by two different chemical dispersion methods. For that, nine different chemically soil samples were collected from Department of Córdoba and Sucre on the Colombian Caribbean region and were analyzed by Bouyoucos and pipette methods. $(NaPO_3)_6/Na_2CO_3$ solution (Calgon) and a mixture of 0.1 M sodium pyrophosphate solution at pH 10 and Calgon in relation 2:1 were used as chemical dispersants. Results were analyzed using Leite and Oliviera (2002) test and correlation analysis. A significant statistic difference between pipette method and Boyoucos method using the proposed chemical dispersant was found for sand and clay fractions with values of 0.97 and 0.89, respectively. The lower sand contents were obtained using the pipette method with an average value of 26%, followed by proposed dispersant with a value of 28%. Average contents of clay were 44 and 42% by proposed dispersant and the pipette method, respectively.

Key words: Granulometric analysis, dispersants, correlation tests, Bouyoucos.

Resumen

Para cuantificar la composición porcentual real de las fracciones texturales en los suelos es necesaria la individualización de las partículas. El objetivo de esta investigación fue comparar los porcentajes de fracciones granulométricas obtenidas con dos métodos de dispersión química en nueve muestras de suelos recolectadas en los departamentos de Córdoba y Sucre en la región Caribe colombiana, utilizando los métodos de Bouyoucos y pipeta. Como dispersantes se usaron soluciones de $(NaPO_3)_6/Na_2CO_3$ (calgón) y una mezcla propuesta constituida por pirofosfato de sodio 0.1M a pH 10 y calgón en una relación 2:1. Los resultados se analizaron utilizando el test de Leite y Oliveira (2002) además de pruebas de correlación. Se encontró relación significativa entre ambos métodos utilizando la mezcla propuesta para las fracciones de arena y arcilla, con valores de 0.97 y 0.89, respectivamente. Los menores contenidos de arena (26%) se obtuvieron utilizando el método de pipeta, seguido del dispersante propuesto con un valor de 28%. Los contenidos de arcilla promedios fueron 44 y 42% por el uso del dispersante propuesto y el método de pipeta.

Palabras clave: Análisis granulométrico, dispersantes, pruebas de correlación.

Introduction

The distribution of particle size fractions in the soil directly influences the dynamics of water, aeration and management practices, and allows also to know the resistance and cohesion of the particles. Chakraborty *et al.* (2006) consider that the size analysis is a necessary practice in physical measurements of soil and are frequently used in the textural classification; plus the contents of sand, silt and clay are the basis for estimating the hydraulic properties and are essential for the identification and classification of soil (Tomasella *et al.*, 2000; Silva dos Santos *et al.*, 2008).

Common methods for soil size analyzes are Bouyoucos method or hydrometer and pipette. According Gee and Or (2002), besides these there are other methods such as pressure sensor, X-ray and laser diffraction; in all cases the results depend on the method of determination.

According Gee and Bauder (1986) the analysis of soil by hydrometer method is based on the relationship between the deposition rate and the soil particle diameter, it is easy to implement and allows fast and reliable measurements. According Zobeck (2004) the particle size distribution refers to the different kinds of particles that compose the soil.

Although the pipette method ensures a greater precision, complexities in assembling the technique and the measuring time have limited their use. Beuselinck *et al.* (1998) indicate that this method is time and labor consuming and the results depend on the technique used in the laboratory and the accuracy of the work of the analyst. Leon (2001) found that the method that comes closest in precision to the pipette is the method of Bouyoucos.

The Geographical Institute Agustín Codazi (IGAC) (IGAC, 2006) established among its methodologies, besides the determination of texture fractions with the pipette and Bouyoucos methods, the use of chemical dispersants ($(NaPO_3)_6 + Na_2CO_3$) called calgon and the slow and fast shaking despite of the dispersant calgon, which gives as result variations in the amount of the sand, silt and clay fractions in the same soil sample. Jorge *et al.* (1985) state that the homogenization of the methods of analysis makes easy the comparison and interpretation of results obtained by different institutions across the country.

The efficient separation of soil aggregates depends on chemical dispersants, efficiency, and the relationship between the ions in the dispersing solution and the permanent and pH dependent charges of the clay fraction. Mauri *et al.* (2011) indicates that for a soil suspension that is effectively dispersed is necessary to replace flocculants cations like Al^{3+}, Ca^{2+}, Mg^{2+}, that frequently saturate the clay, by monovalent cations of larger hydrated ionic radios that follow the sequence in decreasing efficiency order: $Li^+ > Na^+ > K^+ > Rb^+ > Cs^+$. Searching for proposing an acceptable methodology for soil laboratories, the aim of this study was to compare the Bouyoucos and pipette methods in the analysis particle content of sand, silt and clay using two solutions of chemical dispersants.

Materials and methods

The research was performed at the Laboratory of Soil and Water of the Faculty of Agricultural Sciences of the Universidad de Cordoba, Colombia, in nine soil samples collected in the departments of Cordoba and Sucre. For the chemical and mineralogical characterizations of the soil the protocols established by the IGAC were used (IGAC, 2006). To get the granulometric fractions, 60 g of soil from each soil that was previously dried, grinded and sieved in 2 mm sieve were used and placed in plastic containers were each one was mixed with 10 ml of chemical dispersant plus 400 ml of water. The chemical dispersants used were calgon ($(NaPO_3)_6 + Na_2CO_3$) and a mix of sodium pyrophosphate ($Na_4P_2O_7$) 0.1M pH 10 and ($(NaPO_3)_6 + Na_2CO_3$)) (calgon) in a 2:1 proportion.

Soil samples were placed on 600 ml plastic containers where the dispersant and water were slowly added. These containers were left to rest for 24 h before being subjected to mechanical shaking on a shaker at 60 rpm for 6 h and to determination of the granulometric fractions by the Bouyoucos method (IGAC, 2006). For the textural analysis of soil by the pipette method, the subsamples were sent to IGAC.

To compare the chemical dispersants the protocol proposed by Leite and Oliveira (2002) was used. The test, known as L.O., consists on a decision rule built on the basis of the 'F' statistic proposed by Graybill (1976) to evaluate the mean error and in the analysis of the coefficients for lineal correlation. According to the researchers Y_j and Y_i are quantitative vectors, Y_j indicates a method, process or alternative treatment and Y_i is the standard treatment; two methods are considered statistically the same if simultaneously, after the lineal regression adjustment, it is true that: $Yi = \beta_0 + \beta_1 Yi$, $\beta_0 = 0$ and $\beta_1 = 1$, being the value R_{yjyi} very close to 1. Taking into account that this situation is more ideal than real, the authors opted for a modification that applies a more complete statistical analysis.

The proposed modification consisted in performing simultaneously 'F' and 't' tests. Thus, if 'F' (H0) < 'F' tabulated with n-2 degrees of liberty at a ἀ level of significance, the H0 hypothesis is not denied and the intercept is considered to equal as 0 and the slope equals 1. Additionally to the 'F' test, it is important to evaluate the alternative of precision associated to the standard method to quantify the mean error.

To determine the significance of the error a 't' test was performed. In this case, if calculated 't' (mean error 't') < tabulated 't' for n-1 degrees of liberty, the H0 hypothesis is accepted, indicating that the mean error equals 0. In addition, there should be a high coefficient of correlation (close to 1) and 'F' (H0) should not be significant as mean error. However, in the process there is a bias since, even if a high value is found for the coefficient of correlation, this one is relative, because there is a possibility of a high dispersion Y_j in respect to Y_i, thus, a comparison with the expression: $1 - |e|$ is required and observed in Table 1.

From these assumptions, the obtained results are contrasted with the decision rule (Table 1). Where it is indicated that eight scenarios may happen, from which only one is considered to determine equality between the studied or proposed method and the standard method. In addition to this test, the clay, sand and silt data obtained were analyzed by correlations and later classified in textural categories.

Table 1. Decision rules proposed by the Leite and Oliveira test for the significance between methods of particle dispersion.

Scenario	$F(H_0)$	$t_{(e)}$	R_{YjYi}	Decision
1	ns	ns	$R_{yjyi} \geq (1-\|e\|)$	Yj=Yi
2	ns	ns	$R_{yjyi} \leq (1-\|e\|)$	Yj≠Yi
3		*	$R_{yjyi} \geq (1-\|e\|)$	Yj≠Yi
4	ns	*	$R_{yjyi} \leq (1-\|e\|)$	Yj≠Yi
5	*	ns	$R_{yjyi} \geq (1-\|e\|)$	Yj≠Yi
6	*	ns	$R_{yjyi} \leq (1-\|e\|)$	Yj≠Yi
7	*	*	$R_{yjyi} \geq (1-\|e\|)$	Yj≠Yi
8	*	*	$R_{yjyi} \leq (1-\|e\|)$	Yj≠Yi

*: Significant at 5% for the t and F tests. ns: no significant for the t and F tests. $|e|$: mean error. R_{YjYi}: Coefficient of correlation between the analyzed methods. Yj: standard method. Yi: alternative or proposed method.

Results and discussion

The characterization analysis (Table 1) showed that the soil pH was variable between 4.7 and 8.0, organic matter (OM) between 0.5 and 2.9%; calcium between 0.4 and 87.4, magnesium between 0.1 and 10.0 and exchangeable acidity between 0.40 and 6.20 $cmol_c/kg$.

Mineralogical analysis of clay showed that kaolinite and quartz were the predominant minerals, which demonstrates the extreme chemical poverty and the pedological state of the parental materials, due to the weathering in the places where the soil was taken. The type of kaolinite clay and the high amount of quartz in these soils gives them the low or none expansibility and low cationic exchange capacity. Soils in this study showed the presence of gibbsite, which contributes to charges dependent on pH, high points of zero charge and high degree of clay flocculation that can reduce the effect of the deflocculants ions. Acevedo-Sandoval *et al.* (2004) and Peacock and Rimmer (2000) indicate that the point of zero charge (pzc) is an important property of iron oxides and the interactions among them with clay dependent of pH; at low pH oxides precipitate on the Surface of the clay minerals and once formed, these coats are stable to high pH.

Determination of contents of granulometric fractions

When determining the sand, clay and lime contents it was found that the proposed method – composed solution ($Na_4P_2O_7$) 0.1M pH 10 and $(NaPO_3)_6 + Na_2CO_3$— a lower content of sand was determined tan using the dispersant calgon ($(NaPO_3)_6 + Na_2CO_3$); this happened when the Bouyoucos method was used, indicating an increase in the dispersion of fractions that composed the soil.. Rodrigues *et al.* (2011) found that higher $PO_4^{=}$ concentration in the solution improve the dispersion capacity of clay in soils of order like Oxisols, Mollisols an Alfisols.

When comparing the sand contents with both dispersants by the Bouyoucos and pipette methods (Table 3) the lowest content was obtained with the latest one, indicating that this methodology was the most efficient in the quantification of this fraction, result that was verified by 26% of sand found with this me-

Table 2. Chemical and mineralogical characterization of soil samples collected in Cordoba and Sucre, Colombia.

Soils	pH	MO	Ca	Mg	K	Na	Al+H	ECEC	Clay mineralogy		
	1:1	%	-------------- $cmol_c\ kg^{-1}$ ------------------						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	10.00	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	0.51	0.40	0.1	0.13	0.11	0.40	2.10	—	—	—
Maximum	8.0	2.93	87.4	10.0	1.54	0.4	6.20	90.2	—	—	—
Average	6.2	1.40	23.5	4.10	0.50	0.2	2.00	29.2	—	—	—
SD	1.3	0.90	29.3	4.1	0.50	0.1	2.80	29.1	—	—	—

Clay mineralogy: ++++ Dominant (>50%); +++ Abundant (30-50%); ++ Common (15-30%); + Present (5-15%); tr =Trace (<5%). S.D. Standard deviation.

thod, compared to 28 and 32% obtained with the mix proposed with the authors and calgon when using the Bouyoucus method. These results are explained if the chemical properties of Na and its dispersant properties are taken into account as demonstrated by Corá *et al.* (2009) when finding that this element reduces the osmotic pressure of the suspension and contributes with the dispersion of aggregates present in the soil. Moreover, the concentration of the element was higher in the dispersant mix proposed, which increases the pH up to 9.1 in the suspension soil:water:dispersant, allowing a higher separation of the aggregates (Seta and Karathanasis, 1996).

Table 3. Comparison between the sand and clay contents by the Bouyoucos method vs. Pippete method using calgon or mix.

Sample No.	Sand (%)			Clay (%)		
	Calgon	Mix	Pippete	Calgon	Mix	Pippete
1	29	25	18	37	43	57
2	25	18	16	51	66	63
3	15	13	7	33	38	40
4	67	64	68	16	18	14
5	55	51	55	22	25	19
6	17	15	10	35	38	32
7	24	21	17	50	56	54
8	30	22	29	53	53	39
9	27	23	15	48	55	61
Average	32	28	26	39	44	42
SD	17.6	17.6	21.2	13.3	15.6	18.1
Minimum	15	13	7	16	18	14
Maximum	67	64	68	53	66	63

Calgon: $(NaPO_3)_6 + Na_2CO_3$. Mix: $Na_4P_2O_7 + ((NaPO_3)_6 + Na_2CO_3)$ in ratio 2:1

The clay contents determined with the dispersants by the Bouyoucos method vs. the pipette method were 44% with $[Na_4P_2O_7 + (NaPO_3)_6 + Na_2CO_3]$, 42% with pipette and 39% with $(NaPO_3)_6 + Na_2CO_3$. According to Donagemma *et al.* (2003) the increase in the percentage of the clay fraction is an indicative of a higher efficiency of the dispersion treatment used by the reduction in the proportion of pseudocomponents, specially silt. This results can also be explained by the high contents of Na in the $[(NaPO_3)_6 + Na_2CO_3 + Na_4P_2O_7]$ that allows substitution of Al and Ca cations that normally saturate the exchange sites and favor flocculation. According to Ruiz (2005) the chemical dispersion is based on the increase in repulsion between the particles, due to the increase in the diffuse double layer by saturation of the complex for cationic exchange with Na, causing precipitation of Al and Ca. De Sousa Neto *et al.* (2009) found that HCl + NaOH at 1.0 mol/l concentration was more efficient in dispersing the clay in soils with high contents of calcium carbonates. According to Spera *et al.* (2008) cations like Na that have high hydration degree, form complexes and increase the distance between particles, thus the short distance attraction forces are not there and the system gets dispersed.

One factor that may have contributed to increase the dispersing ability of textural soil fractions quantitated by the Bouyoucos method and the dispersant proposed + calgon was the use of slow stirring at 60 rpm for a period of 6 h. With this variation in stirring time, particles in the suspension kept a longer friction between them, generating breakdown of aggregates and hence the granulometric separation of particles that are flocculated. Kettler *et al.* (2001) state that mechanical stirring is used in combination with chemical treatments to help dispersion of soil particles. Silva dos Santos *et al.* (2010) found that slow stirring gives lower variation coefficients, additionally to more precise data when compared with fast agitation methods.

For sand and clay fractions was observed that the mix [$Na_4P_2O_7$ + $(NaPO_3)_6$ + Na_2CO_3], using slow stirring, resulted in an important methodology for physical and chemical dispersion to determine the soil texture, additionally it is simpler and faster than the one used to determine the fraction for the pipette method.

Coefficients of correlation for the sand percentage (Figures 1a and 1b) show a significant correlation (r = 0.97, P < 0.05) between the obtained values with the studied methods, indicating a high confidence between the pipette method using calgon and the alternative dispersion method vs. the Bouyoucos method. Similar results are observed when comparing the pipette method vs. the Bouyoucos method (r = 0.98).

For the clay fraction (Figure 1c and 1d) the coefficient of correlation between the dispersion method proposed vs. the pipette was r = 0.89 (P < 0.05) indicating that, independently from the used method the results are similar. Contrarily, when comparing the pipette method vs. Bouyoucos + calgon no statistical difference was detected (r = 0.81, P > 0.05). For sand and clay contents the dispersant calgon at 60 rpm the efficiency was less than with the proposed method, however, the Bouyoucos method using calgon as dispersant is considered as standard for most of the labs in the country (IGAC, 2006).

Dispersant capability of the solution in the proposed method, including the treatment at 60 rpm, is favored by the increase in pH in the suspension up to 9.2, which triggers an increased zeta potential, where positive and negative charges, thus electrostatic repulsion forces, are very weak and allow greater repulsion between the particles and therefore a widening in the diffuse double layer and better dispersion of the particle size fractions. According to Spera *et al.* (2008) the thickness of the diffuse double layer is an important and critical factor influencing the dispersion of particles and flocculation; Grohmann and Mitchell (1996) consider stability in suspension for clay dispersion requires high pH for an increase of negative charges in the suspension.

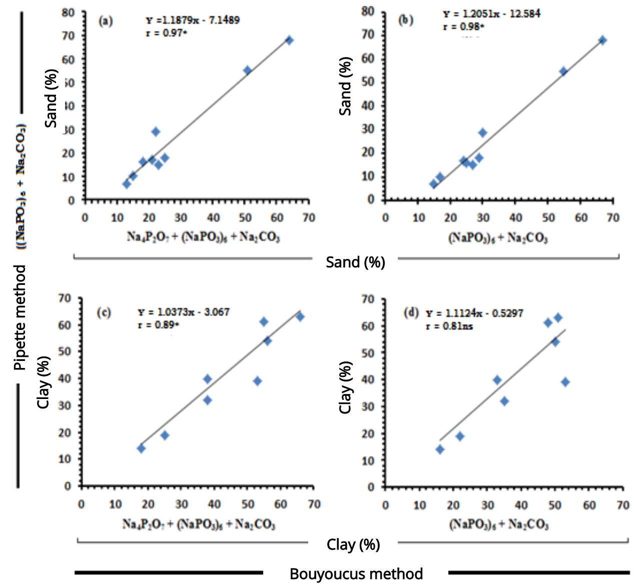

Figure 1. Correlation between the sand and clay percentages obtained with the pipette method vs. the methods of chemical dispersion.

a and b: correlations % sand; c and d: correlations % clay. * P < 0.05, ns no significant.

When the test Leite de Oliveira was applied to analyze the results of the percentages of sand and clay obtained by the three methods for each soil, it was found that there is identity between methods when the results of sand content obtained are compared by the pipette method with the calgon dispersant and Bouyoucos method using the proposed mix as a dispersant (Table 4) mixture. These results are explained since the 'F' and 't' tests did not show significant differences, with coefficients of correlations 0.95 and 0.98, respectively. A similar situation happened for the clay content, with coefficients of correlation of 0.67 and 0.93. According to Akiyoshi and Dalvan (2009) the biggest problem of dispersion is associated with the clay fraction, because when these particles are not dispersed could result in aggregates that underestimate its clay content and overestimate the silt content.

Table 4. Identity of the chemical dispersion methods in the quantification of sand and clay according to the decision test by Leite and Oliveira.

Y_j	Y_i	R^2	$F(H_0)$	$t_{(e)}$	$R_{YjYi} \geq 1-\|e\|$	Decision
			Sand			
$(NaPO_3)_6Na_2CO_3$	$(NaPO_3)_6 + Na_2CO_3\ Na_4P_2O_7$	0.98	14.18^*	5.36^*	Yes	$Y_j \neq Y_i$
Pipette	$(NaPO_3)_6 + Na_2CO_3\ Na_4P_2O_7$	0.95	2.49^{ns}	1.68^{ns}	Yes	$Y_j = Y_i$
Pipette	$(NaPO_3)_6 + Na_2CO_3$	0.97	17.26^*	3.99^*	Yes	$Y_j \neq Y_i$
			Clay			
$(NaPO_3)_6Na_2CO_3$	$(NaPO_3)_6 + Na_2CO_3\ Na_4P_2O_7$	0.93	7.45^*	5.07^*	Yes	$Y_j \neq Y_i$
Pipette	$(NaPO_3)_6 + Na_2CO_3\ Na_4P_2O_7$	0.79	0.15^{ns}	0.85^{ns}	No	$Y_j \neq Y_i$
Pipette	$(NaPO_3)_6 + Na_2CO_3$	0.67	0.52^{ns}	0.85^{ns}	No	$Y_j \neq Y_i$

*: Significant at 5% for the 't' and 'F' tests. ns: no significant for the 't' and 'F' tests.

The results of this study indicate that the values of the statistical tests did not show significant differences between the methodologies (Bouyoucos with calgon and the proposed dispersant) used at 60 rpm vs. the pipette methodology. Therefore, the only obstacle for identity in the methods is the low coefficient of regression.

Table 5. Percentages of sand, clay and lime by Bouyoucos and texture classifications obtained by different methodologies in nine soils of Córdoba y Sucre, Colombia.

Soils	Methodology[a]	Sand	Clay	Silt	Texture[b]
	Calgon[a]	28	38	34	f- ar
1	Mix[b]	25	43	32	ar
	Pipette	18	57	25	Ar
	Calgon	25	52	24	ar
2	Mix	17	66	16	ar
	Pipette	16	63	21	ar
	Calgon	14	34	52	f- ar- li
3	Mix	12	39	49	f- ar- li
	Pipette	7	40	53	f- ar- li
	Calgon	67	16	17	f -a
4	Mix	64	18	18	f -a
	Pipette	68	14	18	f –a
	Calgon	55	22	22	f -a
5	Mix	51	25	24	f -a
	Pipette	55	19	26	f –a
	Calgon	16	35	49	f- ar- li
6	Mix	15	38	47	f- ar- li
	Pipette	10	32	58	f- ar- li
	Calgon	24	50	26	ar
7	Mix	21	56	23	ar
	Pipette	17	54	29	ar
	Calgon	30	53	17	ar
8	Mix	22	53	25	f- ar
	Pipette	29	39	32	f- ar
	Calgon	27	48	25	ar
9	Mix	22	55	22	ar
	Pipette	15	61	24	ar

a. Bouyoucos method with chemical dispersant $(NaPO_3)_6$ + Na_2CO_3 (calgon) and $NaPO_3)_6 + Na_2CO_3\ Na_4P_2O_7$ (proposed mix). b. Textures: f-ar: clay loam; ar: arcilloso; f-ar-li: silty clay loam; f-a: sandy loam.

Finally, when analyzing the sand, clay and silt percentages quantified by the two chemical dispersants at 60 rpm using the Bouyoucos method and the ones determined by the pipette method and calgon, it is observed that the texture classification is equivalent in most of the soils (Table 5). Chaudhari *et al.* (2008) concluded that with the Bouyoucos method using calgon as dispersant the contents of clay are higher than with the pipette method, in this way significant differences are present and avoid agreement on the texture analysis. Vitorino *et al.* (2007) consider that the granulometrical analysis need enough energy to break the binding forces of the aggregates and humic substances, which need to be fragmented by mechanical shocks. According to Tavares and Magalhães (2008) the total dispersion of the soil agrees with the destruction of aggregates. When comparing the textural classes by the proposed mix — $(NaPO_3)_6$ + Na_2CO_3+ $Na_4P_2O_7$— it is noticeable that there are no different textures from the ones obtained by pipette, which does not happen with the Bouyoucos method with the dispersant calgon, in which different textures were defined for soils 1 an 8.

Conclusions

The proposed mix as chemical dispersant reduces the sand percentages and increases the clay percentages.

Between the proposed mix and the pipette method identity in methods was found when evaluating the sand fraction.

A significant relationship between the clay fraction obtained with the dispersant ($(NaPO_3)_6$ + Na_2CO_3 + $Na_4P_2O_7$) and the pipette method was found, however, with the identity test the same results were not obtained.

Using the proposed mix the same texture classification tan using the pipette method.

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