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Determination of Total Aluminium, Iron and Silicon in Soils under Rubber by Acid Dissolution Technique

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Soil samples were decomposed by acid dissolution in acid digestion bombs and the sample solutions analysed by atomic absorption spectrophotometry. Analytical results of aluminium, iron and silicon with mean coefficients of variation of 2.1%, 4.7% and 5.8%, respectively, showed that the procedure can be used for the determination of total aluminium, iron and silicon in Malaysian soils. Percentage recovery of aluminium, iron and silicon added to the soils and soil digests were well above 90% thus indicating the high level of accuracy and precision of the method.

The procedure for the determination of total elemental content of soil materials consists of two steps, the first involves the decomposition of the material and the second concerns the analysis of the elements from the breakdown of the soil matrix. The decomposition of soil materials can either be done by fusion techniques using a variety of flux mixtures or acid dissolution in pressure vessels[1-6]. Once the sample is brought into solution, the elemental constituents are determined by the normal atomic absorption spectrophotometric method. Refinements in atomic absorption spectrophotometry reported by Price and Whiteside[5] and Boar and Ingram[7] permit the accurate determination of the main constituents, Al, Fe and Si.

Presently, the Rubber Research Institute of Malaysia (RRIM)[8] adopts the method of decomposing soil materials by sodium carbonate fusion at 1000°C. Both Al and Fe in the solution are determined by atomic absorption spectrophotometry while Si is analysed by gravimetry. The analytical techniques, though practical, are tedious as they involve several steps. It is on this consideration that attempts were made to look for a more efficient and reliable method for routine analysis. This paper gives the Al, Fe and Si contents of ten soils determined by acid dissolution in an acid-digestion bomb followed by elemental analysis by atomic absorption spectrophotometry. The quality of the results and the suitability of the method for analysis of soil materials are studied and discussed.

MATERIALS AND METHODS

Different soil types (*Table 1*) at 0–45 cm depth were sampled in bulk from soil profile pits located in various areas under rubber. The soil samples were oven-dried at 55°C, ground to pass through a sieve (< 2 mm size) and sub-sampled for chemical analysis[8]. Some of their properties are shown in *Table 1*. For elemental analysis of Al, Fe and Si, each of the soil samples was further ground to a fine powder (< 60 mesh).

Decomposition of Soil Material in an Acid-digestion Bomb

The method of Price and Whiteside[5] was modified and used to obtain an acid solution of the soil material; 200 mg 60-mesh size soil, 5 ml water, 2 ml aqua regia (containing one part of concentrated nitric acid to three parts of concentrated hydrochloric acid) and 1 ml 40% hydrofluoric acid were introduced into the

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Teflon cup in an acid-digestion bomb (Parr Instrument Company; Model No. 4745-A238Ac and capacity 25 ml). The digestion bomb was closed by hand-tightening the screw cap containing the Teflon sealing disk, heated in an oven at 135°C ±2°C for 30 min and then allowed to cool to room temperature. On cooling, about 10 ml of 4% boric acid was added to the digest in the vessel and re-heated as before for 20 min. The final digest was cooled, transferred into a 100 ml polypropylene volumetric flask and made up to the mark. In subsequent analysis of the soil digest, polypropylene containers instead of glass flasks were used.

Analysis of Soil Digest

The concentrations of Al, Fe and Si in the soil digests were determined by atomic absorption spectrophotometry on an IL Video 22 Unit. An air-acetylene flame was used for Fe and a nitrous oxide-acetylene flame for Al and Si. The stock solutions for calibration graphs were prepared from British Drug House Co. Ltd. 'Spectrosol' grade reagents. Various reagents like boric acid, hydrofluoric acid and 2% potassium chloride solution were added to the standards to compensate for those present in the sample solution.

Aluminium. An aliquot of the soil digest with 5 ml of 2% potassium chloride solution as an ionisation buffer was transferred to a 100 ml standard flask and made up to the mark with distilled water. The concentration of Al in the diluted solution was determined with the atomic absorption spectrophotometer (AAS) set at a wavelength of 396.2 nm and previously calibrated with standard Al solutions of range 0–100 μg Al/ml solution.

Iron. An aliquot of the digest was diluted and passed through the AAS set at wavelength of 372.0 nm and calibrated with Fe standard solutions of concentrations 0–10 μg Fe/ml solution. The concentration of the Fe was determined from the calibration graph.

Silicon. An aliquot of the soil digest was diluted and passed through the AAS set at a wavelength of 251.6 nm and calibrated with Si standard solutions of concentrations 0–100 μg Si/ml solution. The concentration of Si was determined as for Al and Fe.

The determination of Al, Fe and Si content in each of the soil types was done in six replicates. In each replicate, a separate soil sample was used. The elemental contents of the soils were expressed as oxides of the elements and in microgramme per gramme oven-dry material.

Recovery of Al, Fe and Si added to the soil. To each of the six soils, known quantities of Al, Fe and Si prepared from standard solutions and equivalent to about 50% of the inherent Al, Fe and Si content in the soils were added. The composition of Al, Fe and Si in the soils was determined as before. The percentage recovery of the element added was calculated from the elemental content of the treated and untreated soil.

Recovery of Al, Fe and Si added to the soil digest. To each of the freshly prepared soil digests in the Teflon cups, known quantities of Al, Fe and Si were added. The digest was then analysed for Al, Fe and Si. The same digest without the addition of Al, Fe and Si was also analysed. The percentage recovery of the elements was determined from the two sets of results.

RESULTS

The ten soils described in *Table 1* represent a cross-section of all soil types in Peninsular Malaysia. The highly weathered soils are acidic with pH values varying from 3.8 to 4.5. Singh[10] and Noordin[11] had shown that the soils contain mainly Al_2O_3, Fe_2O_3 and SiO_2 and the clay mineral is predominantly kaolinite (> 50%). The soils also have different textural properties. The Selangor, Segamat, Kuantan, Batu Anam and Durian series soils have combined silt and clay fractions of about 90% by weight while those of Holyrood and Serdang series soils are less than 21% by weight. Total organic carbon content in the soils varies from 0.78 g/kg to 2.23 g/kg soil. The marine clay of the Selangor series soil has the largest amount of organic carbon content.

Mean values of Al_2O_3, Fe_2O_3 and SiO_2 together with the standard deviation (sd) and

TABLE 1. SOME CHEMICAL AND PHYSICAL PROPERTIES OF THE SOILS

Soil series	Sub-group[a] (parent material)	Texture[b] Coarse sand	Fine sand	Silt	Clay	pH[c]	Organic[d] carbon (g/kg soil)
Batu Anam	Aquic Paleudult (shale)	0.5	7.4	32.9	59.2	4.4	1.31
Durian	Plinthaquic Paleudult (shale)	9.9	13.7	31.6	44.8	3.9	1 24
Holyrood	Typic Kandiudult (sub-recent alluvium)	62.8	23.2	3.4	10.6	4 6	0.98
Kuantan	Typic Kandiudox (basalt)	5.6	7 3	27.1	60.0	4.5	1.94
Malacca	Xanthic Hapludox (schist)	13 4	10.9	11 8	63.9	4.4	1.82
Munchong	Typic Hapludox (schist)	6.4	14.7	29.0	49.9	4.2	1.75
Rengam	Typic Kandiudult (granite)	38.2	9.5	9.8	42.5	4.0	1 27
Segamat	Typic Kandiudox (andesite)	2.2	3.1	18.3	76.4	4.3	1 59
Selangor	Aeric Tropic Fluaquent (marine clay)	0.8	9.6	41.1	48.5	3 8	2.23
Serdang	Typic Kandiudult (sandstone)	43.1	36.3	4.2	16.4	4.3	0.78

[a]According to Keys to Soil Taxonomy by Soil Survey Staff - USDA (1989)
[b]Expressed as percentage of oven-dry soil and determined by Pipette Method[9]
[c]Measured with a pH meter on a suspension of soil in distilled water at ratio 2:5
[d]Determined by the Walkley and Black's titration method

coefficients of variations (cv) are shown in *Table 2*. In most soils, SiO_2 content was the highest followed by Al_2O_3 and Fe_2O_3 contents. Relatively, soils of Kuantan, Malacca, Munchong and Segamat series have higher Al_2O_3 and Fe_2O_3 contents and soils of Batu Anam, Durian and Selangor series have higher SiO_2 content. The variation of analytical results for Al_2O_3, Fe_2O_3, and SiO_2 differed with mean cv values of 1.89%, 4.25% and 5.32%, respectively. In the Fe_2O_3 results, the cv values for Rengam series soil were well above 5.0%. As for SiO_2, cv of three soils, namely the Holyrood, Rengam and Serdang series soils, were well above 5.0%.

To determine the reliability of the acid-digestion bomb method, a recovery study was initiated and the results presented in *Table 3*. The average recoveries for Al, Fe and Si in the six soils were 97.2%, 94.4% and 98.5%, respectively. Of the determinations, there were five cases (two for Al, one for Fe and two for Si) where the element recovered was more than what was added. However, the values were confined to a narrow range of 102.6% to 107.0%. There were two cases of poor recovery (< 90%), *viz*., one for Al and one for Fe.

Using the same approach, the atomic absorption spectrophotometric method was

TABLE 2. ALUMINIUM, IRON AND SILICON CONTENT OF THE SOILS

Soil series	Al_2O_3 (g/kg soil)			Fe_2O_3 (g/kg soil)			SiO_2 (g/kg soil)		
	Mean[a]	s.d.	c.v.	Mean[a]	s.d.	c.v.	Mean[a]	s.d.	c.v.
Batu Anam	189.0	2.6	1.4	34.4	1.8	5.2	452.8	22.7	5.0
Durian	288.6	2.3	0.8	50.6	2.7	5.3	501.5	27.4	5.5
Holyrood	213.2	2.2	1.0	49.4	2.5	5.1	370.2	34.9	9.4
Kuantan	556.9	6.5	1.2	418.4	8.3	2.0	204.4	2.1	1.0
Malacca	510.2	11.4	2.2	266.9	3.6	1.4	273.1	15.8	5.8
Munchong	611.7	9.0	1.5	202.6	5.3	2.6	230.7	6.0	2.6
Rengam	175.5	7.6	4.3	60.6	5.4	8.9	360.2	27.2	7.5
Segamat	423.6	6.4	1.5	241.7	4.4	1.8	379.0	19.1	5.0
Selangor	320.0	4.3	1.4	48.1	1.7	3.5	504.7	16.6	3.3
Serdang	172.2	6.3	3.7	69.3	4.0	5.8	341.5	27.3	8.0
Mean	346.1		2.1	144.2		4.7	361.8		5.8

[a]Mean of six replicates
s.d. = standard deviation
c.v. = coefficient of variation (%)

evaluated by determining the percentage recoveries of Al, Fe or Si added to the final soil digests consisting of different elemental composition (*Table 4*). For Al, Fe and Si, mean recoveries were high with values of 98.5%, 99.5% and 95.0%, respectively. There were seven cases in which the percentage recovery exceeded 100.0% (ranged from 100.4% to 105.4%). Barring these results, the recoveries were generally good with values well above 90.0%.

DISCUSSION

From the results described above, the acid-digestion bomb method can be applied for the determination of Al, Fe and Si in Malaysian soils.

The coefficients of variations, though ranging from 0.79% to 9.42%, were satisfactory when compared with the corresponding range of values, 3.62% to 57.50%, for soil chemical parameters reported by Zainab[12]. The average recovery for the three elements in the soils and soil digests was well above 90% indicating that the acid digestion step was complete with little loss of materials and the atomic absorption spectrophotometric method for elemental determination gave a high level of accuracy.

The procedure using aqua regia and hydrofluoric acid to dissolve the soil material in the digestion bomb is simple and straightforward. A heating temperature of 135°C was found to be sufficient for complete dissolution, although temperatures of 150°C and 110°C had been reported by Price and Whiteside[5] and Bernas[3], respectively. The presence of varying amounts of organic matter in the soils had no serious limitations on the acid-digestion method and the subsequent elemental analysis by atomic absorption spectrophotometry as the amount of acids used was more than adequate to destroy all the organic matter. Contamination arising from the use of hydrofluoric acid is avoided as the activity of fluorides in the digest is eliminated with the addition of excess boric acid. Further, all reagents and extracts were prepared and stored in polypropylene containers. With

TABLE 3. RECOVERIES OF ALUMINIUM, IRON AND SILICON ADDED TO THE SOILS[a]

Soil series	Al_2O_3			Fe_2O_3			SiO_2		
	Added	Found[b]	Recovery (%)	Added	Found[b]	Recovery (%)	Added	Found[b]	Recovery (%)
Batu Anam	94.8	88.05	93.2	20.01	17.15	85.7	213.90	210.26	98.3
Durian	132.27	129.59	98.0	20.01	18.30	91.5	213.90	226.31	105.8
Kuantan	283.43	292.12	103.1	20.01	19.30	96.5	106.95	103.53	96.8
Munchong	283.43	252.82	89.2	100.07	103.78	103.7	106.95	99.68	93.2
Segamat	188.95	202.18	107.0	114.36	110.64	96.7	192.51	181.60	94.3
Selangor	151.16	139.82	92.5	22.87	21.16	92.5	256.68	263.31	102.6
Mean			97.2			94.4			98.5

[a]Mean of six determinations. Amount added (in g/kg soil) corresponds to about 50% of Al, Fe and Si in the soils.
[b]*After adjusting for values in the control (without soil sample)*

TABLE 4 RECOVERIES OF ALUMINIUM IRON AND SILICON ADDED TO THE SOIL DIGESTS[a]

Sample no	Al_2O_3 (µg)				Fe_2O_3 (µg)				SiO_2 (µg)			
	Present	Added	Found	Recovery (%)	Present	Added	Found	Recovery (%)	Pressent	Added	Found	Recovery (%)
1	7 22	7 56	14 44	95 5	1 00	1 43	2 37	95 8	11 34	6 42	17 73	99 5
2	12 81	7 56	20 07	95 9	3 95	5 72	9 98	105 4	5 69	6 42	11 64	92 7
3	9 33	7 56	16 63	96 4	4 77	5 72	10 78	105 1	8 02	6 42	14 22	96 6
4	10 92	7 56	18 21	96 4	5 35	5 72	11 38	105 4	6 18	6 42	12 06	91 6
5	12 09	7 56	19 39	96 5	8 29	5 72	13 84	97 0	4 90	6 42	10 80	91 9
6	4 12	7 56	11 75	101 0	1 43	1 43	2 77	93 7	7 98	6 42	13 95	93 0
7	4 27	7 56	11 90	100 9	1 20	1 43	2 57	95 8	8 13	6 42	14 07	92 5
8	5 03	7 56	12 55	99 5	1 00	1 43	2 40	97 9	7 66	6 42	13 63	93 0
9	6 54	7 56	14 06	99 5	1 03	1 43	2 43	97 9	10 33	6 42	16 66	98 6
10	4 46	7 56	12 24	102 9	0 71	1 43	2 14	100 4	9 20	6 42	15 57	99 2
Mean				98 5				99 5				95 0

[a]Mean of six determinations

rapid improvement in the usage of atomic spectroscopy, a wider range of elements can be determined from the same soil digest without extra labour and time.

The method of acid dissolution in a digestion bomb appears to be suitable for the determination of Al, Fe and Si in Malaysian soils. The method does not require a temperature of 1000°C, platinum crucibles which are expensive and have to be kept in a safe at all times and fusing agents which are often used in excess of the sample weight by five- to ten-fold.

CONCLUSION

Total Al, Fe and Si in Malaysian soils can be determined by acid dissolution of the soil matrix in an acid-digestion bomb followed by determination of the elements by atomic absorption spectrophotometry. The method is simple and straightforward compared with the existing fusion method. With further refinements, a wider range of elements other than Al, Fe and Si can be determined from the same digest.

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