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Technical-Grade Chemical-Assisted Extraction of Humic Substances from Wet Decanter Solids for Soil Amelioration in Short-Term Incubation Test

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ABSTRACT

The development of smart and sustainable agriculture requires low-cost soil amendment technologies derived from agro-industrial residues. This study evaluated the use of technical-grade extractants, caustic soda (NaOH), monosodium glutamate (MSG), and urea for extracting humic substances (HS) from wet decanter solids (WDS) and assessed their potential as soil ameliorants for Ultisols. A completely randomized design (CRD) with three extractant concentrations (0.1, 0.5, and 1.0 M) and three replications was applied during a 24 h extraction period. Among the treatments, 1.0 M NaOH produced the highest HS yield (2.29%) and C-HS content (1.28%), along with a high cation exchange capacity (CEC) [172.40 cmol(+) kg^{-1}] and an alkaline pH (12.27), indicating abundant phenolic and carboxylic functional groups as well as enriched nutrient content. Application of NaOH-extracted HS at 8,000 mg L^{-1} significantly improved the chemical properties of Ultisols, including increases in soil pH (+4.07), electrical conductivity (EC, +1.31 dS m^{-1}), organic matter (OM, +9.80%), CEC [+20.6 cmol(+) kg^{-1}], and nutrient availability (+4.42% C, +0.27% N, +34.50 ppm, and +0.41 cmol(+) kg^{-1} exchangeable K) compared with the control. These findings indicate that HS extracted with technical-grade 1M NaOH from WDS is an economical and easy-to-develop organic additive that supports waste utilization, improves soil function at concentration levels of 6000-8000 mg L^{-1} (can increase pH and P availability), and promotes sustainable agriculture based on circular bioeconomy.

1. INTRODUCTION

Soil is the main foundation for supporting food security because nearly 95% of human food needs come from fertile soil [1, 2]. Good soil quality, characterized by organic matter (OM) content and a balance of physical, chemical, and biological properties, is key to sustainable agricultural productivity. However, intensive agricultural practices that rely heavily on chemical fertilizers have caused serious land degradation. In Indonesia, an estimated 14 million hectares of land have been degraded due to declining OM content, soil structure damage, and loss of soil microbial diversity [3]. Long-term use of inorganic fertilizers can cause a decrease in soil pH, salt accumulation, and the death of decomposing microorganisms that play an important role in maintaining the nutrient cycle. The capacity of the soil to provide nutrients for plants has decreased, which in turn threatens national agricultural productivity. Therefore, more sustainable and appropriate soil management strategies are needed in the application of agroecology, as well as a circular economy approach that reuses organic waste as a soil ameliorant.

One of the main components of soil organic matter (SOM) that plays an important role in maintaining soil quality is humus. Humus is the complex decomposition of OM that is rich in active functional groups, such as carboxylates and phenols, which make it highly reactive to various chemical processes in the soil [4, 5]. The presence of humus has been proven to increase cation exchange capacity (CEC) due to its negative charge, enabling it to bind and retain cations that are important nutrients for plants. The complex structure of humus also plays a role in improving soil aggregation, increasing water retention, and maintaining soil structure stability to make it more resistant to erosion [6]. Humic substances (HS) can function as biostimulants, which are chemicals like auxins and cytokinins that promote plant development by boosting enzyme activity, absorbing nutrients, and controlling hormone balance [7, 8]. By promoting wider plant resilience, HS additionally fortifies plant defenses against biotic and abiotic metabolic stress [9, 10]. By enhancing soil quality and promoting organic plant development, HS serves as a multipurpose agent that promotes the sustainability of agricultural systems.

The growing need for HS in agriculture, new extraction and purification technologies from organic sources are being developed. Traditionally, HS is derived from natural resources such as peat, lignite, and leonardite, which have high and stable characteristics [11, 12]. However, the scarcity of these resources, as well as the environmental consequences of mining and utilizing them, has prompted the quest for new raw materials that are more abundant, cheaper, and ecologically acceptable. Organic agricultural and agro-industrial waste have a high lignocellulose content and may be humified to produce stable humus [13, 14]. The use of this organic waste not only improves the availability of fresh potential HS but also aligns with the concepts of sustainable agriculture and a circular economy, minimizing pollution and enhancing soil fertility. Developing techniques to extract HS from alternative sources is an important step toward more effective and ecologically sustainable agricultural production.

Indonesia is the world's top palm oil producer, with crude palm oil (CPO) output expected to reach 3,828 million tons in 2025. In January 2025, CPO and palm kernel oil (PKO) output totaled 4,184 million tons. Wet decanter solid (WDS), a solid residue produced by oil separation using a centrifugal decanter, is one of the agro-industrial wastes produced during CPO processing. WDS has a high lignocellulose content as well as nutrients including C, N, P, K, and fiber, making it a promising lignocellulosic biomass for a variety of applications, including HS [15]. The WDS is frequently underused and, if not properly controlled, has the potential to pollute the environment. WDS is hydrophobic and difficult to degrade spontaneously due to its high water content and residual oil [16]. If not managed properly, WDS can pollute the surrounding environment, especially through water and soil pollution. The anaerobic decomposition and humification processes of lignocellulose components in WDS can produce HS that have various benefits for the soil, such as increasing CEC, improving soil structure, increasing water retention, and acting as a biostimulant for plant growth [17]. The WDS has the potential to be used as a raw material for the production of activated carbon, which is also a form of HS with high heavy metal adsorption capacity [18, 19]. Therefore, the development of methods for extracting and purifying HS from WDS is a strategic step in supporting sustainable agriculture and environmentally friendly agro-industrial waste management, as well as producing high agronomic value products.

Extraction of HS from organic materials, including WDS, is generally carried out using alkaline solutions such as sodium hydroxide (NaOH). These alkaline solutions serve to dissolve the humic fraction from the lignocellulose matrix through saponification and dissolution of active functional groups [20]. Although this approach is efficient in obtaining HS, the extraction efficiency is frequently low due to limited alkali penetration into the complex lignocellulose structure. The use of pro-analytical (p.a.) NaOH might raise production costs, induce equipment corrosion, and require careful alkali waste management to prevent environmental pollution [21]. Aside from efficiency and cost concerns, the quality of the HS produced is also a problem. Conventional extraction of HS can result in uneven molecular weight, inadequate functional group content, and low biostimulant activity [22]. As a result, technical-grade extractant innovation is required to provide high-quality HS with decreased production costs and little environmental impact. The technical-grade extractants' potential from monosodium glutamate (MSG) and chemical fertilizers such as urea.

A knowledge gap remains in the development of wet decanter-based HS due to the lack of integration of extractant optimization studies at the technical level with the assessment of the agronomic efficacy of the resulting product. Most studies only use extraction yield indicators and molecular characterization as metrics of process success, but the impact of various extractants on the efficacy of HS in improving the chemical characteristics of Ultisols has not been thoroughly assessed. Variations in extractants can provide different functional group compositions and humification levels, which can affect the ability of logam complexation, increase CEC, and increase nutrient availability in Ultisols. Therefore, research is needed to evaluate the efficacy of various technical-grade extractants in producing optimal HS in the process and most beneficial in improving Ultisols quality. This is a novel method that may enhance the efficacy of HS extraction. The selection of these compounds takes into account not only technical benefits but also factors of availability and cost. The NaOH, MSG, and urea are cost-effective, easily accessible in the market, and applicable to farmers. Consequently, this methodology may yield a more cost-effective, practical, and eco-friendly HS extraction technology. This study has examined the potential technical-grade from NaOH, MSG, and urea for the extraction of HS from WDS and its application on Ultisols.

2. MATERIAL AND METHODS

This research was conducted from May 2025 to August 2025 at the Laboratory of the Department of Soil Science and Land Resources, Faculty of Agriculture, Andalas University, Padang, West Sumatra, Indonesia. The X-ray fluorescence (XRF), Fourier transform infrared (FTIR), and Scanning Electron Microscope-Energy Dispersive X-ray (SEM-EDX) analyses were conducted at the Andalas University Central Laboratory, Padang, West Sumatra, Indonesia.

2.1 Experimental design

This study used a completely randomized design (CRD) with three replicates in two experiments. Stage 1st investigated the characteristics of HS from wet decanter solids (WDS) against the potential of technical-grade extractants (NaOH, monosodium glutamate, urea), and three concentrations (0.1, 0.5, and 1 M) for 24 hours. Meanwhile, Stage 2nd examined the effects of HS from selected extracts on changes in soil surface charge and nutrients in an oil palm plantation, with varying application rates: (A) 0, (B) 2000, (C) 4000, (D) 6000, and (E) 8000 mg L^{-1}. Experimental units for both experiments were randomly assigned to each treatment level to ensure unbiased and robust statistical analysis.

2.2 Technical-grade extractants

The extractant used was sodium hydroxide (NaOH), technical-grade with a purity level of approximately 95–98%. Meanwhile, MSG of Ajinomoto is a white crystal with a composition of ± 99% MSG, ± 0.5–1% water, and < 0.1–0.5% impurities (NaCl, free glutamic acid, sulfate ash). Single chemical fertilizer, namely Urea [$CO(NH_2)_2$], is in the form of white crystalline granules with a nitrogen content of approximately 46% N in the form of amide. Potential of technical-grade extractants based on concentration level and pH (Table 1).

2.3 Humic substances production

The main raw material for HS is WDS collected from PT. Bina Pratama Sakato Jaya Inc (Table 1). The WDS is dried at 70 ℃ for 2 × 24 hours and weighed for the HS production process under experiment 1st. The HS (fulvic + humic acid) production batch uses 5g of dried WDS per sample with an extractant ratio of 1:5 w/v at three concentrations and three replicates for consistency and standardization of the experiment. The incubation process was carried out for 24 hours and centrifuged at a speed of 4000 rpm for 30 minutes. Next, it was filtered using Whatman grade 42 filter paper, and the resulting filtrate was dried at 70 ℃ for 1 × 24 hours. The WDS and HS (fulvic + humic acid) were analyzed characteristically in the laboratory.

Table 1. Descriptive statistics of chemical characteristics from wet decanter solids (WDS)

Analysis		Unit	Min	Max	Mean	SE	SD
Proximate	Moisture	% (w/w)	78.00	82.67	79.62	1.52	2.64
	Volatile matter		76.18	83.40	79.79	2.08	3.61
	Ash		19.72	22.50	21.11	0.80	1.39
	Fixed carbon		4.09	4.41	4.26	0.09	0.16
pH	H_2O	-	7.00	7.20	7.10	0.06	0.10
	KCl 1 M		6.80	7.00	6.93	0.07	0.12
	PZC*		6.60	6.90	6.77	0.09	0.15
EC		dS m^{-1}	2.00	2.00	2.00	0.00	0.00
CEC		cmol(+) kg^{-1}	45.00	47.00	46.00	0.58	1.00
Main Nutrients	OC	% (w/w)	18.61	22.88	20.74	1.23	2.14
	Total N		0.28	0.31	0.29	0.01	0.02
	C/N ratio	unit	60.03	81.71	71.94	6.35	11.00
	Available P	ppm P_2O_5	934.96	1042.53	988.74	31.05	53.79
	K-exch	cmol(+) kg^{-1}	13.30	13.38	13.34	0.02	0.04
	C/P ratio	unit	0.0200	0.0220	0.0210	0.0006	0.0010
	N/P ratio	unit	0.00027	0.00033	0.00029	0.00002	0.00003
Organic pollution	COD	mg O_2 kg^{-1}	136.92	163.93	150.43	7.80	13.51
	BOD		41.81	46.54	44.18	1.37	2.37
	COD/BOD ratio	unit	2.94	3.92	3.42	0.28	0.49
	Total oil content	% (w/w)	12.25	13.60	12.93	0.39	0.68

Note: PZC* = point of zero charge with formula [(2 × pH KCl) – pH H_2O]; EC = electrical conductivity; CEC = cation exchange capacity; OC = organic C; COD = chemical oxygen demand; BOD = biochemical oxygen demand; SE = standard error; SD = standard deviation; and n = 3 samples.

2.4 Soil sampling

Soil samples were collected from a forested area within an oil palm plantation landscape in Sijunjung–Dharmasraya, West Sumatra, Indonesia (101°24'13.52'' to 101°32'6.80'' E and 0°47'48.97'' to 1°2'39.73'' S). The soils were classified as Ultisols according to the USDA Soil Taxonomy system. Surface soil samples (0–20 cm) were collected using a composite sampling approach, in which five subsamples obtained from each of five sampling points arranged in a zigzag pattern were thoroughly homogenized to produce a representative composite sample of the study site. The selected area represents a long-standing oil palm plantation ecosystem, characterized by highly weathered, acidic soils with low nutrient availability. The soil samples were then taken to the laboratory for processing by (a) drying, (b) crushing and screening with a 2 mm sieve, and (c) thorough mixing and weighing according to the experiment. Soil was used for experiment II to assess the effect of selected HS on soil surface charge with different rates (0 to 8000 mg L^{-1}). Soil was weighed as much as 100 g absolute dry equivalent, and HS-NaOH-WDS was weighed according to the dose given under experiment 2nd. Soil samples and HS-NaOH from WDS following the dose were homogenized by adding the amount of water according to the field capacity of the soil in the experimental pot manually until well mixed. The samples were then incubated for one week and analyzed in the laboratory.

2.5 Data analysis

The WDS and HS analysis used guidelines issued by the Ministry of Agriculture (MOR) decision standard No. 261/KPTS/SR.310/M/4/2019 concerning minimum technical requirements for soil ameliorants, namely pH (H_2O, KCl 1 M), and pH-PZC [23], electrical conductivity (EC), CEC, organic and inorganic C, total N, and oxide composition with PANalytical Epsilon 3XL Benchtop XRF Spectrometer. The characteristics of organic pollution in WDS were evaluated by determining chemical oxygen demand (COD), biological oxygen demand (BOD), and total oil content (TOC) to assess organic load, residual oil content, biodegradability, and potential environmental impacts. COD was measured using a closed reflux colorimetric method (APHA 5220D), while BOD was determined following a standard incubation procedure (APHA 5210B). TOC was quantified by Soxhlet extraction of n-hexane based on a modified gravimetric method (EPA 1664A). Functional group analysis with IRT Racer 100 FTIR Shimadzu A217061 also supports the potential of HS for application as a soil ameliorant. SEM-EDX is capable of magnifying up to 1,000,000 times (on the Carl Zeiss EVO 10 type), which is combined with Energy Dispersive X-ray Spectroscopy (EDX) as a representation of the distribution of these elements that can be visualized in the form of line scans and mapping [24].

Soil analysis was focused on soil surface charge characteristics and nutrients, namely pH (H_2O, KCl 1M, and PZC), EC, mineral and OM composition, CEC, and nutrients such as organic C, total N, available P, and exchangeable K [25]. Ameliorant and soil analysis were subjected to statistical analysis using the software of Microsoft Excel 2016 and SPSS 23. The statistical analysis used was the analysis of variance (ANOVA) and Duncan's multiple range test (DMRT). After detecting a significant effect with ANOVA, DMRT was used as a post hoc test to identify which specific group means differ from each other. Significance levels were determined as follows: if the calculated F value exceeded the tabulated F value at the 5% level, the result was considered significant and marked with "*"; if it exceeded the tabulated F value at the 1% level, the result was considered highly significant and marked with "**". The "*" and "**" annotations indicate these significance levels.

3. RESULT AND DISCUSSION

3.1 The characteristics of wet decanter solids

Descriptive statistics of the chemical characteristics of WDS indicate that this material is a carbon-rich and highly organic residue with great potential for processing into HS in Table 1. The moisture content ranged from 78.07% to 82.67%, with an average value of 79.62%, indicating that WDS is a semi-solid biomass produced immediately after the decanter separation process. This value is in accordance with previous studies on palm oil decanter dregs, which reported moisture contents between 76 and 80%, confirming that WDS retains a significant amount of water and readily biodegradable OM. The high volatile matter content (79.79%) and low fixed carbon content (4.26%) further indicate the dominance of labile organic compounds compared to the poorly biodegradable carbon fraction. The ash content (21.11%) reflects the presence of mineral elements derived from palm oil fruit residues and processing impurities. The WDS has a composition favorable for biological transformation and humification processes.

The pH values measured in water (7.10) indicate a near-neutral chemical environment, which generally favors microbial activity and nutrient cycling during composting and soil application. Furthermore, the point of zero charge (PZC) of 6.77 is very close to the measured pH, indicating that the surface charge characteristics of WDS are highly sensitive to small pH fluctuations. This behavior may affect the adsorption and release of nutrients and dissolved organic compounds during decomposition. EC remains low and stable at 2.00 dS m^{-1}, indicating a relatively low dissolved salt concentration and reducing the risk of salinity stress when applied to agricultural soils. Furthermore, the average CEC was 46.00 cmol(+) kg^{-1}, indicating a substantial nutrient retention capacity. High CEC values are generally associated with the abundance of oxygen-containing functional groups, such as carboxyl and phenolic groups, which are precursors of HS and contribute significantly to improving soil fertility.

The organic C of WDS reached 20.74%, confirming its role as a source of carbon-rich biomass. However, the total nitrogen content was relatively low (0.29%), resulting in a very high C/N ratio of 71.94. This ratio is well above the optimum range for rapid microbial decomposition, suggesting that WDS alone may experience nitrogen limitation during biological stabilization. Nevertheless, materials with high C/N ratios are often considered valuable feedstocks for humification processes because they provide an abundant carbon substrate for the formation of stable humic macromolecules [26]. Therefore, co-composting with nitrogen-rich materials may be necessary to accelerate mineralization and improve process efficiency while maintaining the potential for high humic yields [27].

Available P of WDS ranged from 934.96 to 1042.53 ppm P_2O_5, with a mean of 988.74 ppm. This relatively high phosphorus concentration indicates that WDS contains a large reserve of plant-available P derived from residual oil palm fruit tissue and microbial mineralization processes occurring during palm oil processing. Phosphorus is an essential macronutrient involved in energy transfer, nucleic acid synthesis, and root development [28]. Therefore, its abundance increases the potential value of WDS as a nutrient-rich organic material. K-exch showed very low variability, ranging from 13.30 to 13.38 cmol(+) kg^{-1} with an average of 13.34 cmol(+) kg^{-1}. Such high levels of exchangeable K are characteristic of palm oil residues because oil palm biomass accumulates large amounts of potassium during growth. Potassium plays a crucial role in enzyme activation, osmotic regulation, photosynthetic transport, and stress tolerance in plants [29]. The high and stable K concentrations observed in WDS suggest that it can serve as an effective supplemental potassium source for agricultural soils, particularly in highly weathered tropical soils where K depletion often limits crop productivity.

Oxygen demand further revealed a substantial organic load in WDS. The average COD was 150.43 mg O_2 kg^{-1}, while the average BOD was 44.18 mg O_2 kg^{-1}. These values indicate the presence of a significant amount of oxidizable organic compounds, including carbohydrates, proteins, lipids, and lignocellulosic constituents. The COD/BOD ratio ranged from 2.94 to 3.92, with an average value of 3.42. This ratio indicates moderate biodegradability, meaning the WDS contains readily degradable substrates and a more resistant organic fraction. From a bioconversion perspective, this characteristic is advantageous because it favors gradual transformation into stable humified OM rather than rapid mineralization. Comparable COD and BOD values have been reported for palm oil decanter residue, which has been characterized as a highly biodegradable and organic-rich byproduct of the palm oil milling process [30].

One important characteristic of WDS is its high residual oil content. The average TOC was 12.93%, ranging from 12.25 to 13.60%. This finding is highly consistent with previous reports showing residual oil concentrations of approximately 12–16% in palm oil decanter dregs. The substantial retention of the oil fraction after CPO extraction indicates that WDS remains an important secondary resource containing recoverable lipids and energy-rich compounds. Residual oil can positively contribute to the formation of HS through oxidative polymerization reactions and can also increase the calorific value of the material for bioenergy applications. However, excessive oil concentrations can inhibit microbial degradation and should be considered when designing composting or humification strategies. The physicochemical profile indicates that WDS is characterized by high water content, high organic C, substantial CEC, moderate biodegradability, and significant residual oil concentration. The combination of high organic carbon (20.74%), high CEC (46 cmol(+) kg^{-1}), moderate COD/BOD ratio (3.42), and substantial residual oil content (12.93%) indicates that WDS has strong potential as a feedstock for HS extraction, organic fertilizer production, and circular bioeconomy applications. However, the very high C/N ratio suggests that N supplementation may be necessary to optimize biological stabilization and maximize nutrient recovery efficiency. These findings collectively support the utilization of WDS as a value-added resource rather than a discarded waste stream in sustainable palm oil production systems.

3.2 The characteristics of humic substances from wet decanter solids

The type and quantity of technical-grade extractant had a significant impact on the extraction performance of HS from WDS, demonstrating that solvent chemistry is important in freeing the humified organic fraction from palm oil residues (Table 2). NaOH consistently outperformed the other extractants tested. Increasing the NaOH concentration from 0.1 to 1.0 M raised the HS production from 1.33% to 2.29%, with carbon-bonded HS (C-HS) rising from 0.75% to 1.28% (Table 1). This enhanced performance was due to the very alkaline environment (pH > 11), which promoted carboxyl and phenolic group ionization, increased humic solubility, and broke organomineral linkages in the lignocellulosic matrix. Similar alkaline extraction processes have been shown to effectively dissolve humic fractions from compost and biomass-derived materials [31]. However, increased alkalinity lowers the remaining humin proportion, indicating that insoluble and resistant OM is converted into soluble humin forms.

MSG also demonstrated high extraction capability, with HS rising from 1.26% to 2.12% as concentration increased. It remained lower than NaOH. This activity may be explained by the presence of carboxyl and amine functional groups in MSG, which may decrease organomineral connections and partially solubilize HS via chelation and hydrogen bonding interactions [32]. In contrast, urea demonstrated the lowest extraction efficiency, providing just 0.51-0.81% HS. The majority of the organic portion remained humin, suggesting urea's poor ability to ionize aromatic-carboxyl compounds. Rather than acting as a solvent, urea is largely a nitrogen donor that aids in the following humification process [33]. Based on the overall extraction performance, technical grade NaOH with a concentration of 1.0 M was identified as the most effective selected concentration for recovering HS from WDS by producing the highest HS and C-HS yields and was therefore selected for further characterization and evaluation (Table 2). However, MSG is a promising, environmentally friendly alternative with moderate extraction efficiency, while urea is more suitable as an additional N enrichment agent during the humification process rather than as the main extractant.

Table 2. The potential of technical-grade extractant solutions is based on the difference in concentration of HS in the wet decanter solids (WDS)

Potential of Technical-Grade Extractants	Molarity (M), g mol^{-1}	g L^{-1} (w/v)	pH Extractants	pH of HS (1:5 w/v)	C-HS, %	HS, %
NaOH	0.1	4	11.97	11.93	0.75	1.33
	0.5	20	12.30	12.27	1.01	1.80
	1	40	12.43	12.33	1.28	2.29
Monosodium Glutamate	0.1	16	7.20	5.80	0.70	1.26
	0.5	84	7.40	6.27	0.71	1.27
	1	168	7.57	6.53	1.18	2.12
Urea Fertilizer	0.1	8	6.30	5.23	0.28	0.51
	0.5	32	6.40	5.33	0.30	0.54
	1	60	6.60	5.73	0.45	0.81

Note: HS = humic substances; C = carbon, and n = 27 samples.

Table 3. Effect of technical-grade extractants by concentration on the quality of humic substances (HS) from the wet decanter solid

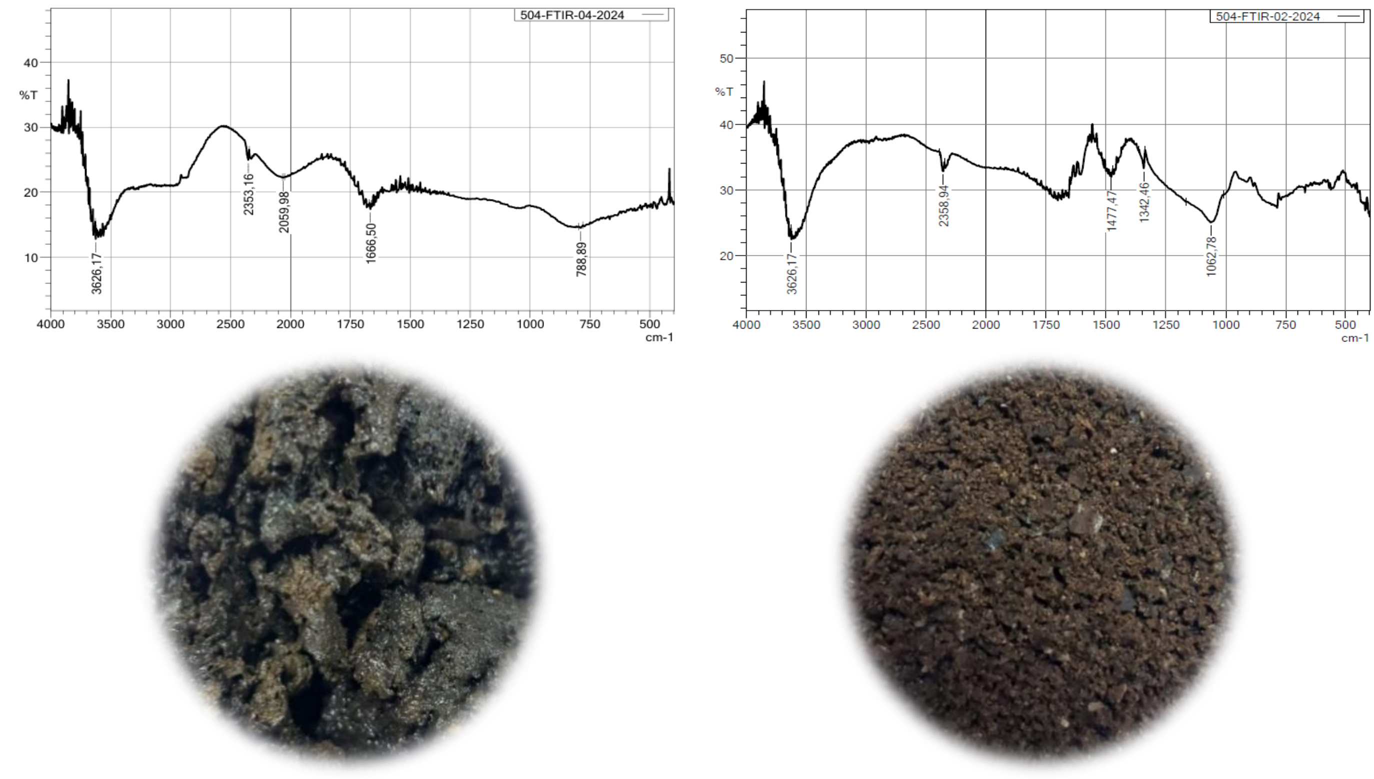
Potential of Technical-Grade Extractants	pH			EC	CEC	Total N
	H_2O	KCl 1 M	PZC	dS m^{-1}	cmol(+) kg^{-1}	%
NaOH 1 M	12.00 a	11.70 a	11.40 a	2.00	172.40 a	0.56 c
MSG 1 M	8.40 b	7.60 b	6.80 b	2.00	168.00 b	6.64 b
Urea 1 M	7.70 c	7.30 c	6.90 b	2.00	155.20 c	10.72 a
CV (%) - Duncan's Test	*1.07***	*1.13***	*1.54***	-	*1.23***	*1.00***
SE	*0.08*	*0.08*	*0.11*	-	*1.67*	*0.03*

Note: Numbers in the same column followed by the same lowercase letter are not significantly (ns) different according to the Duncan's test at the 5% (*) and 1% (**) level; MSG = monosodium glutamat; PZC = point of zero charge with formula [(2 × pH KCl) – pH H_2O]; EC = electrical conductivity; CEC = cation exchange capacity; CV = coefficient of variation; SE = standard error and n = 9 samples.

The physicochemical quality of extracted HS is also highly influenced by the kind of extractant. Extraction selected with concentration 1 M of NaOH resulted in the greatest pH (pH H_2O 12.00; pH KCl 11.70; PZC 11.40) and CEC of 172.40 cmol(+) kg^{-1}, showing significant enrichment of negatively charged functional groups (Table 3). High alkalinity allows carboxyl and phenolic groups to be deprotonated, enhancing exchangeable charge density and colloidal reactivity [5]. Although the overall N content in HS extracted with NaOH was modest (0.56%), its high CEC implies a great potential for nutrient retention and soil development. The HS extracted with MSG had a lower pH but a very high CEC (168.00 cmol(+) kg^{-1}) and a substantially greater total N (6.64%), indicating the incorporation of nitrogen from glutamate. The HS extract with urea had the greatest total N (10.72%) but the lowest CEC (155.20 cmol(+) kg^{-1}), indicating that urea enriches nitrogen rather than increasing humic charge characteristics. EC values above 2 dS m^{-1} in all treatments indicate high ion content from functional groups and residual salts. Thus, NaOH remains the most efficient extractant for humic function, whereas MSG and urea may give additional nutritional advantages.

The FTIR spectra of WDS and HS extracted using 1 M technical NaOH show significant chemical transformations associated with humification and selective dissolution of OM (Figure 1). The spectra reveal both the preservation of characteristic organic functional groups and the appearance of more oxidized structures after alkaline extraction, confirming the successful conversion of WDS into HS. A broad and intense absorption band centered around 3626 cm^{-1} was observed in both materials, which corresponds to the stretching vibrations of hydroxyl groups (–OH) originating from phenols, alcohols, carboxylic acids, and hydrogen-bonded water molecules. This band is characteristic of HS and reflects the abundance of oxygen-containing functional groups that contribute to CEC and metal complexation. The persistence of this peak after extraction indicates that the hydroxyl-rich aromatic and aliphatic structures are retained in the humic fraction. However, the observed increase in width and intensity in the extracted material indicates increased hydrogen bonding and greater molecular complexity, which are typical characteristics of mature HS.

In the 2358–2360 cm^{-1} region, weak absorption bands were detected in both spectra. These bands are generally associated with O=C=O stretching vibrations associated with carbon dioxide or adsorbed carbonate species. The persistence of these signals after alkali treatment may indicate an interaction between atmospheric CO_2 and the alkali-extracted organic matrix, resulting in the formation of carbonate-related structures. Similar observations have been reported in humic extracts obtained by NaOH extraction, where carbonate species are incorporated into supramolecular humic assemblies [34]. Important transformations occur in the carbonyl region. The WDS spectrum shows a distinct absorption band near 1666 cm^{-1}, which corresponds to the C=O stretching vibration of conjugated ketones, quinones, amides, and carboxyl groups. After NaOH extraction, this band becomes less pronounced and shifts toward lower wavenumbers, while new absorptions appear around 1477 cm^{-1} and 1342 cm^{-1}. These changes indicate structural rearrangements involving oxidation and deprotonation of organic molecules during alkali extraction. The band around 1477 cm^{-1} is associated with aromatic C=C stretching and asymmetric stretching of the carboxyl group (COO^-), while the peak at 1342 cm^{-1} is associated with phenolic O–H bending and symmetric COO^- stretching vibrations. The appearance and intensification of these bands provide strong evidence for the enrichment of carboxylic and phenolic functional groups, which are fundamental structural components of HS and are responsible for their high reactivity in soil systems [35].



(a) (b)

Figure 1. Fourier transform infrared (FTIR) spectra of (a) wet decanter solids (WDS) and (b) humic substances (HS) extracted using 1 M NaOH (technical grade)

The most significant spectral changes were observed in the fingerprint region, specifically at 1062 cm^{-1}. This absorption is generally associated with the C–O stretching vibrations of polysaccharides, alcohols, ethers, and carboxylic acids, as well as the Si–O stretching vibrations associated with silicate-containing organic-mineral complexes. The prominent intensity of this band in the extracted HS indicates increased exposure of oxygenated functional groups following the solubilization of the lignocellulosic structure by NaOH. This increase indicates that alkali extraction effectively disrupts the cellulose-lignin association and releases a humified organic fraction enriched in oxygenated functionalities. These groups are crucial for nutrient retention, metal chelation, and soil aggregation processes. The weaker absorption observed near 789 cm^{-1} in the HS spectrum can be attributed to out-of-plane aromatic C–H bending vibrations and/or Si–O vibrations of silicate minerals. The persistence of this peak indicates that the aromatic structures and OM associated with the minerals remain integrated within the extracted humic matrix. Aromaticity is often considered an indicator of the degree of humification because HS become increasingly rich in condensed aromatic structures during decomposition and stabilization [36].

The FTIR clearly shows that extraction with technical-grade 1 M NaOH induces substantial chemical modifications in the WDS, leading to the formation of HS rich in hydroxyl, carboxylic, phenolic, and oxygen-containing functional groups. The decrease in lignocellulosic markers and the increase in COO^{-}, C–O, and aromatic functional groups indicate increased molecular oxidation and humification. These structural transformations are consistent with the formation of chemically active HS with high CEC, nutrient complexing ability, and metal binding potential. Consequently, the extracted HS are expected to serve as effective soil amendments capable of increasing nutrient availability, soil aggregation, and carbon stabilization in highly weathered tropical soils such as Ultisols.

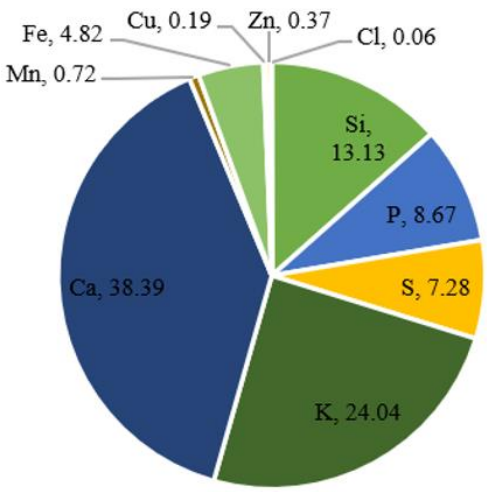


Figure 2. Elemental composition (%) of technical grade 1 M NaOH-extracted humic substances (HS) determined by X-ray fluorescence (XRF) from wet decanter solids (WDS)

The XRF analysis revealed substantial transformations in oxide composition after conversion of WDS into HS by selective extraction using 1 M technical NaOH (Figure 2). After alkali extraction with technical grade 1M NaOH, the elemental profile changed significantly, indicating selective dissolution and concentration of the humified organic fraction. The extracted HS consisted primarily of CaO (38.39%) and K_2O (24.04%), followed by SiO_2 (13.13%), P_2O_5 (8.67%), SO_2 (7.28%), and Fe_3O_4 (4.82%). Small amounts of MgO (0.72%), ZnO (0.37%), CuO (0.19%), and Cl (0.06%) were also identified. The disappearance of carbon as the dominant element detected in the oxide spectrum and the enrichment of mineral-associated elements indicate that the alkali extraction process effectively disrupted organo-mineral complexes and concentrated humic macromolecules associated with exchangeable cations. Calcium enrichment is crucial because Ca^{2+} acts as a bridging ion that stabilizes the supramolecular structure of humic and promotes aggregate formation when applied to soil [5]. Similarly, the substantial increase in potassium and phosphorus concentrations indicates that the extraction process retained nutritionally valuable components, potentially enhancing the agronomic value of the obtained HS.

The high Si (13.13%) in the extracted fraction likely stems from mineral impurities and phytolith residues inherited from oil palm biomass. Silicon has been reported to enhance plant resistance to abiotic stress and contribute to soil structural stability, thereby enhancing the multifunctional benefits of humic amendments [37]. The presence of sulfur, iron, manganese, zinc, and copper further indicates that the extracted HS has the capacity to complex micronutrients through chelation mechanisms. The observed enrichment of nutrient carriers after extraction indicates that technical-grade NaOH not only facilitates the recovery of the humic fraction but also concentrates mineral constituents beneficial for soil fertility management. The XRF indicates that the conversion of WDS to HS through selective extraction with technical-grade 1 M NaOH substantially altered the elemental composition from a residue dominated by organic carbon to a humic-rich material associated with nutrients and minerals. This compositional shift confirms the effectiveness of alkali extraction in recovering functional HS while enriching them with agronomically important nutrients. Therefore, the resulting product has significant potential as a multifunctional soil ameliorant capable of enhancing nutrient retention, CEC, soil aggregation, and long-term soil carbon stabilization, particularly in highly weathered tropical soils such as Ultisols.

The SEM-EDX characterization revealed substantial morphological and elemental transformations following the conversion of WDS into HS by selective extraction with technical-grade 1 M NaOH (Figure 3). The SEM micrographs of the raw WDS revealed a compact, irregular, and heterogeneous surface structure characterized by agglomerated organic particles and the formation of a dense matrix (Figure 3(a)). This morphology is typical of palm oil mill solid residues, which contain lignocellulosic fragments, residual oil, microbial biomass, and partially decomposed OM. The relatively compact surface indicates that most of the organic components remain embedded in complex organo-mineral associations, thus limiting the accessibility of the humified fraction.

The EDX spectrum of the crude WDS further confirmed its organic-rich nature, with carbon (49.09%) and oxygen (22.36%) as the dominant elements. These high concentrations reflect the abundance of carbon compounds and oxygen-containing functional groups derived from the lignocellulosic material and residual palm oil constituents. The P (7.21%), Ca (8.52%), Ti (7.51%), Mg (3.80%), and K (1.50%) were also detected, indicating the presence of mineral nutrients and inorganic constituents associated with the biomass matrix. The high carbon content indicates a significant reservoir of humification precursors, while the presence of phosphorus and calcium may contribute to nutrient retention and subsequent organo-mineral interactions during HS formation [38]. After alkali extraction, the morphology of the obtained HS changed significantly (Figure 3(b)). The extracted material exhibited a highly porous and fibrous structure with the appearance of layered and needle-like crystal aggregates scattered on the particle surface. These morphological changes indicate that NaOH effectively disrupts the original lignocellulosic matrix, dissolving the humifiable organic fraction and exposing previously inaccessible internal structures. The increase in surface roughness and the development of porous domains indicate a larger specific surface area and a greater number of reactive functional groups. These structural modifications are characteristic of HS produced by alkaline extraction, where the breakdown of intermolecular associations increases the accessibility of carboxyl, phenolic, and hydroxyl groups responsible for cation exchange and metal complexation [5].

The EDX analysis of the extracted HS revealed significant shifts in elemental composition. Carbon decreased from 49.09% to 25.99%, while oxygen increased substantially from

22.36% to 45.71%. This increase in the O/C ratio indicates increased oxidation and enrichment of oxygen-containing functional groups, which are fundamental characteristics of HS. Higher oxygen content generally reflects a greater concentration of carboxyl and phenolic functional groups, which contribute to increased CEC and metal-binding properties [10]. Sodium (Na) emerged as the major component (24.08%), indicating that the extraction process concentrated organic compounds. This enrichment is crucial because these functional groups contribute to nutrient cycling and enhance the agronomic value of humic amendments [37].

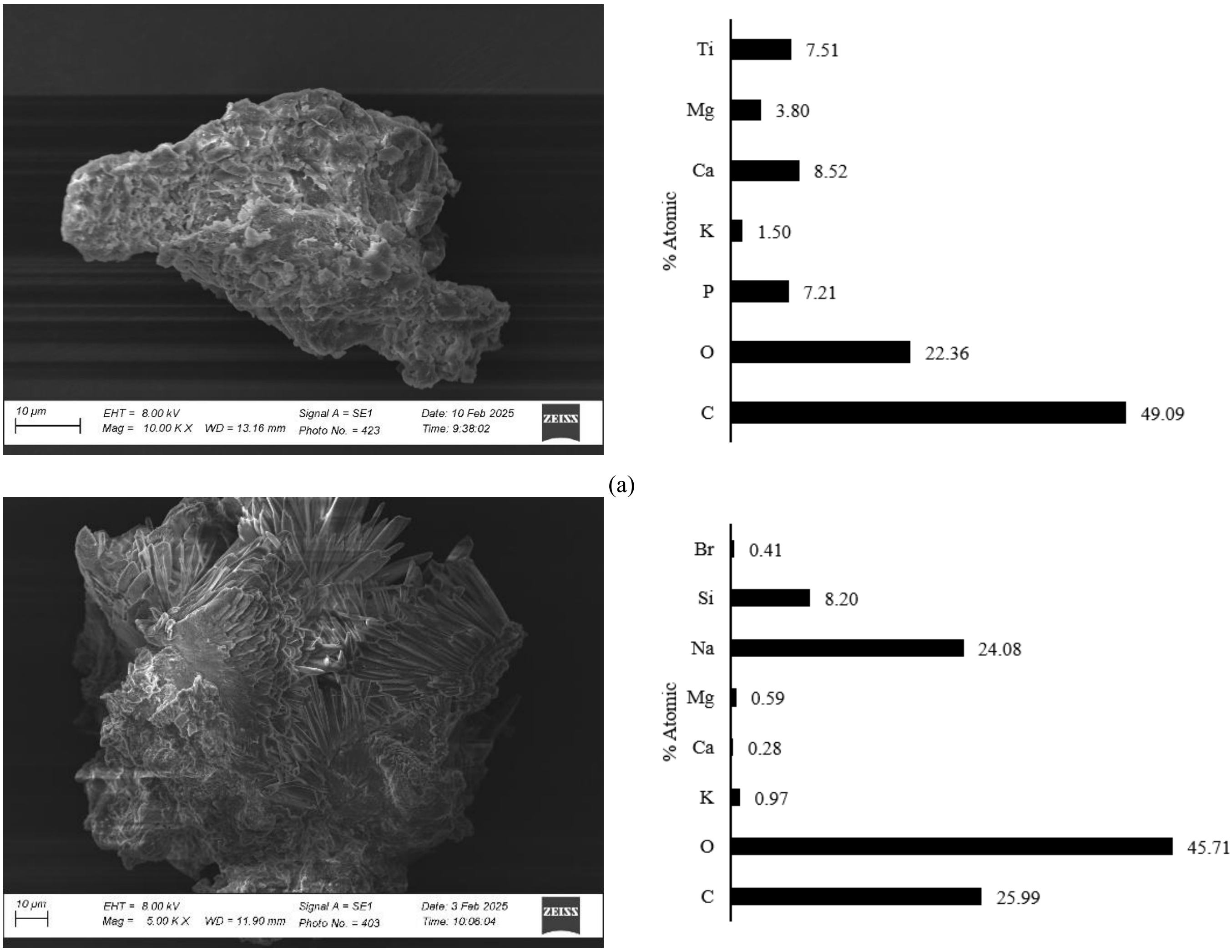


Figure 3. Scanning Electron Microscope-Energy Dispersive X-ray (SEM-EDX) characterization of (a) wet decanter solids (WDS) and (b) humic substances (HS) obtained by extraction with 1 M NaOH (technical grade)

The extracted HS also contained Si (8.20%), Mg (0.59%), K (0.97%), Ca (0.28%), and trace amounts of Br (0.41%). The presence of silicon likely reflects the incorporation of mineral components derived from phytoliths obtained from oil palm biomass. Silicon is known for its role in improving soil physical properties and enhancing plant tolerance to environmental stress. The decreased concentrations of calcium and magnesium compared to the original material suggest partial dissolution or redistribution of these cations during alkaline extraction, while the remaining mineral elements may contribute to the formation of stable organo-mineral complexes. The SEM–EDX provide strong evidence that extraction with 1 M technical NaOH successfully transformed WDS from a solid, carbon-rich residue into a more oxidized and functionally active HS. The significant increase in oxygen and nitrogen levels, coupled with the development of a porous and fibrous microstructure, indicates the formation of HS with improved surface reactivity and nutrient-binding capacity. These physicochemical changes support the potential application of the obtained HS as value-added soil amendments that can increase soil fertility, nutrient availability, and carbon stabilization in sustainable agricultural systems.

3.3 Application of humic substances from wet decanter solids of oil palm waste on Ultisols

The application of HS extracted from WDS using selected technical-grade 1 M NaOH significantly modified the electrochemical properties and surface charge behavior of Ultisols (Table 4). Increasing the HS dosage from 0 to 8000 mg L^{-1} progressively increased soil pH (H_2O) from 4.30 to 8.37 and pH (KCl) from 4.10 to 8.10. This substantial increase in pH indicates that HS effectively neutralizes soil acidity through proton consumption and complexation of exchangeable Al and Fe ions. The humic contains numerous carboxylic (-COOH) and phenolic (-OH) functional groups that undergo deprotonation under alkaline conditions, thereby reducing active acidity and increasing the number of negatively charged sites on soil colloids [39]. These changes were particularly significant in highly weathered Ultisols, where variable-charged minerals dominate the exchange complexes. The observed increase in the PZC from 3.90 in untreated soil to 7.83 at the highest HS dose indicates that HS substantially alters the charge characteristics of soil colloids. In soils with variable charge, PZC represents the pH at which positive and negative charges are balanced on mineral surfaces. The addition of HS promotes the formation of organo-mineral complexes, modifying the electrochemical environment of the Fe and Al oxides that dominate Ultisols. Humic coatings on mineral surfaces can alter proton adsorption and desorption processes, thereby altering surface charge behavior and increasing soil buffering capacity [40]. The significant increase in PZC reflects a strong interaction between humic functional groups and reactive mineral surfaces.

A more important indicator of net surface charge is the relationship between soil pH and PZC. Soil pH remained higher than the corresponding PZC values, indicating that the soil surface carries a net negative charge. The difference between pH(H_2O) and PZC increased from 0.40 units in the control to 0.54 units at 8000 mg L^{-1} HS, indicating a progressive increase in negative charge density. When soil pH exceeds PZC, hydroxyl, carboxyl, and phenolic groups are increasingly deprotonated, resulting in the formation of negatively charged exchange sites. Consequently, the increase in the pH–PZC gap observed in this study indicates that HS promotes the development of an increase in pH-dependent negative charge, which is able to retain nutrient cations and reduce leaching losses.

Table 4. The effect of humic substances (HS) from wet decanter solids (WDS) through selected extraction with technical grade 1 M NaOH on the surface charge of Ultisols

Doses of Humic Substances (mg L^{-1})	**pH (1:1)**			**EC**	**Composition**		**CEC**
	H_2O	**KCl 1 M**	**PZC**		**Mineral**	**OM**	
				dS m^{-1}	**%**		**cmol(+) kg^{-1}**
0	4.30 d	4.10 e	3.90 e	0.35 d	83.80 a	16.20 e	26.60 d
2000	6.70 c	6.40 d	6.10 d	0.36 d	78.40 b	21.60 d	40.60 c
4000	7.63 b	7.17 c	6.70 c	0.74 c	78.20 c	21.80 c	41.20 c
6000	8.20 a	7.77 b	7.33 b	1.36 b	76.40 d	23.60 b	42.93 b
8000	8.37 a	8.10 a	7.83 a	1.66 a	74.00 e	26.00 a	47.20 a
CV (%) - Duncan's Test	*1.51***	*1.09***	*3.11***	*1.04***	*0.06***	*0.23***	*1.53***
SE	*0.09*	*0.06*	*0.16*	*0.01*	*0.04*	*0.04*	*0.49*

Note: Numbers in the same column followed by the same lowercase letter are not significantly different (ns) according to Duncan's test at the 5% (*) and 1% (**) levels; PZC = point of zero charge with formula [(2 × KCl) – H_2O]; EC = electrical conductivity; OM = organic matter; CEC = cation exchange capacity; CV = coefficient of variation; SE = standard error; and n = 15 samples.

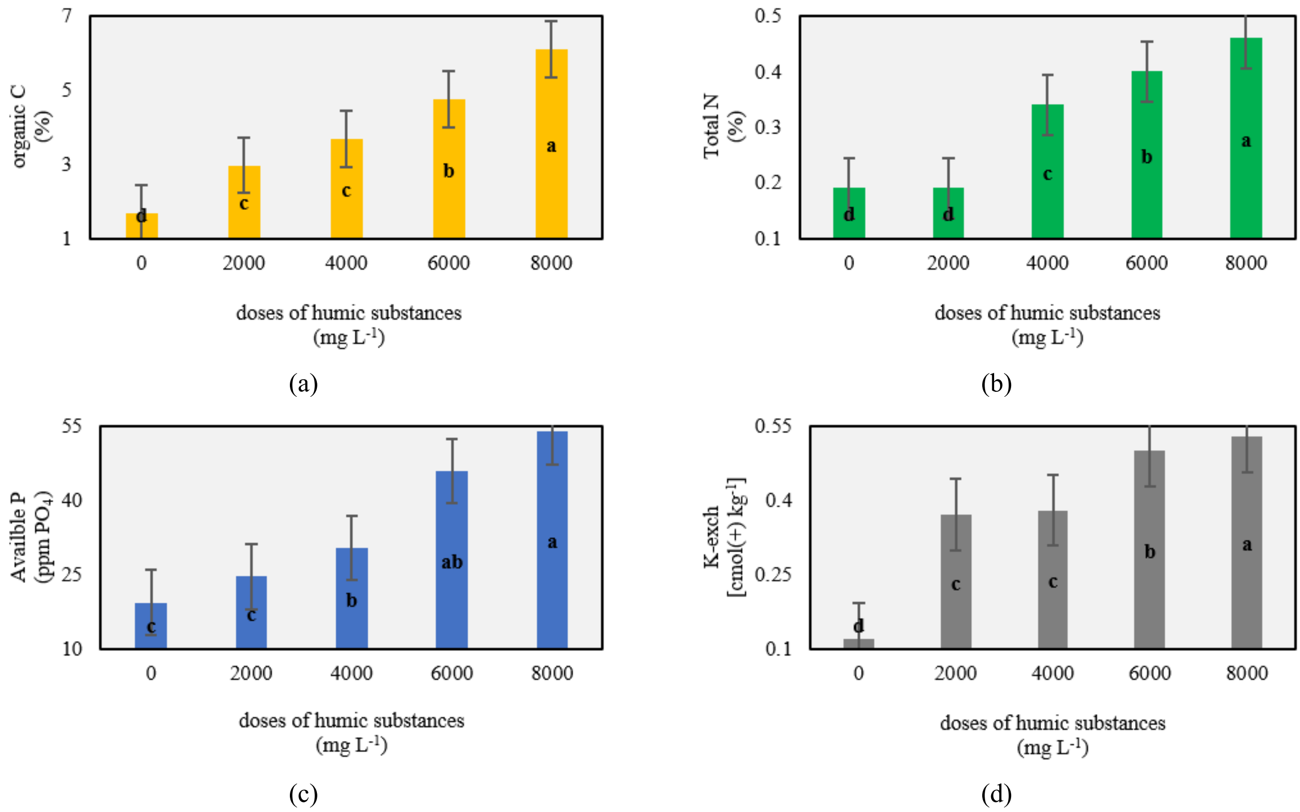


Figure 4. The effect of humic substances (HS) from wet decanter solids (WDS) through selected extraction with technical grade 1 M NaOH on the main nutrient composition: (a) organic C, (b) total N, (c) available P, and (d) K-exch of Ultisols

The increase in EC from 0.35 to 1.66 dS m^{-1} was accompanied by a substantial increase in SOM content from 16.20% to 26.00%, while the mineral fraction decreased from 83.80% to 74.00%. These results indicate that HS were successfully incorporated into the soil matrix and contributed to the accumulation of reactive organic colloids. OM is recognized as one of the most important contributors to CEC due to its high density of dissociable functional groups [41]. A further increase in EC indicates greater availability of dissolved ions in the soil solution, reflecting increased nutrient retention and exchange processes facilitated by HS.

The characteristic increase in surface charge was significant in CEC from 26.60 cmol(+) kg^{-1} in the control to 47.20 cmol(+) kg^{-1} at 8000 mg L^{-1} HS. This represents an increase of approximately 77.4%, demonstrating the effectiveness of HS in generating additional exchange sites. The increase in CEC is mainly due to the dissociation of carboxyl and phenolic groups and the formation of stable organo-mineral complexes that contribute to charge development [42]. Higher CEC values indicate greater nutrient retention, increased buffering capacity, and improved soil fertility. Therefore, HS extracted from WDS acts as a potent modifier of surface charge properties in Ultisols, improving the electrochemical environment through increased negative charge density, increased cation retention, and greater soil chemical stability.

Application of HS extracted from WDS using 1 M technical NaOH significantly improved the nutrient status of Ultisols, as reflected by increases in organic carbon (C), total nitrogen (N), available phosphorus (P), and exchangeable potassium (K-exch) (Figure 4). A clear dose-dependent response, with nutrient concentrations generally increasing with increasing HS application rates from 0 to 8000 mg L^{-1}. These findings demonstrate the ability of HS to improve soil fertility through direct nutrient contributions and indirect enhancement of nutrient retention mechanisms. This response is particularly important in Ultisols, which are typically characterized by low OM content, strong nutrient fixation, and limited nutrient availability due to intensive weathering processes.

The organic C increased substantially from approximately 1.67% in the control to approximately 6.09% with 8000 mg L^{-1} HS (Figure 4(a)). Statistical analysis revealed significant differences between treatments, with the highest dose resulting in the greatest accumulation of soil organic carbon. This increase may be attributed to the direct incorporation of humified carbon compounds derived from WDS. The humic contains stable aromatic and aliphatic carbon structures that resist rapid microbial decomposition and contribute to long-term carbon sequestration [43]. Furthermore, humics promote the formation of aggregates and organo-mineral associations that physically protect SOM from mineralization [4]. The nearly threefold increase in organic carbon observed in this study indicates that extracted HS acts as an effective carbon amendment capable of improving soil quality and carbon storage potential.

A similar trend was observed for total N (Figure 4(b)), which increased from approximately 0.19% in untreated soil to nearly 0.46% at the highest HS application rate. The most pronounced increase occurred between 2000 and 4000 mg L^{-1}, suggesting that a threshold concentration of HS may be required to stimulate nitrogen accumulation. First, HS themselves contain organically bound nitrogen derived from decaying plant debris and microbial biomass. Second, humic can reduce nitrogen loss through ammonium retention and protection of organic N from microbial degradation. Third, increased microbial activity associated with humic addition can enhance nitrogen cycling and mineralization processes [44]. The strong positive relationship between organic C and total N observed in this study suggests that HS application contributed to a simultaneous increase in SOM and nitrogen reserves, thereby enhancing the overall nutrient supply capacity of Ultisols.

Among all the nutrients measured, available P showed one of the most remarkable responses to HS application (Figure 4(c)). Available P increased from approximately 19.32 ppm in the control to over 45.87 ppm 6000 mg L^{-1} HS, and looks the same as the 8000 mg L^{-1} HS (53.82 ppm), representing an increase of nearly ≈180%. Further statistical analysis showed that phosphorus availability increased significantly with increasing HS doses. This response is particularly important in Ultisols because phosphorus is generally immobilized through strong adsorption on Fe and Al oxides. The humic contains numerous carboxylic and phenolic functional groups capable of forming complexes with Fe^{3+} and Al^{3+} ions, thereby reducing phosphate fixation and increasing P availability in the soil solution [45]. Furthermore, humic molecules can compete with phosphate ions for adsorption sites on oxide surfaces, promoting phosphorus desorption and increasing plant accessibility. The significant increase in available P demonstrates the effectiveness of HS in addressing one of the major fertility constraints in highly weathered tropical soils.

The K-exch also responded positively to HS application (Figure 4(d)), increasing from approximately 0.12 cmol(+) kg^{-1} in the control to approximately 0.53 cmol(+) kg^{-1} with 8000 mg L^{-1} of HS. This significant difference indicates that HS application increased potassium retention in the exchange complex. Humic acid has numerous negatively charged functional groups that contribute to CEC, thereby enhancing the soil's ability to retain K^+ ions against leaching losses [46]. Furthermore, the decomposition products of WDS can provide an additional source of potassium, further contributing to the observed increase. The progressive increase in K-exch values with increasing HS dosage indicates that the addition not only provides potassium but also improves the electrochemical environment necessary for nutrient retention and exchange.

The simultaneous increases in organic C, total N, available P, and exchangeable K indicate that HS extracted from WDS functions as a multifunctional soil additive capable of enhancing nutrient supply and nutrient use efficiency in Ultisols. The highest application rate (6000-8000 mg L^{-1}) consistently resulted in the largest increases in all measured parameters, indicating a strong positive relationship between HS concentration and fertility improvement. Mechanistically, these benefits are related to increased OM accumulation, enhanced CEC, reduced phosphorus fixation, enhanced nutrient retention, and enhanced microbial activity. Therefore, the use of HS derived from palm oil processing residues is a promising strategy to improve nutrient-deficient Ultisols while promoting sustainable waste utilization and soil health management in tropical agricultural systems, and promotes a circular bioeconomy model in which palm oil waste is turned into a value-added soil amelioration capable of rebuilding low-fertility tropical soils while reducing industrial waste.

4. CONCLUSIONS

Technical-grade 1 M NaOH (caustic soda) solution was identified as the most effective extractant for recovering HS from WDS, resulting in the highest HS yield (2.29%) and C-HS content (1.28%). The extracted HS showed high CEC [172.40 cmol(+) kg^{-1}] and alkaline pH (12.27), indicating the presence of reactive phenolic and carboxylic functional groups along with macro- and micronutrient enrichment. The application of WDS-derived HS significantly improved the chemical quality of Ultisols, with the 8000 mg L^{-1} resulting in the largest increases in surface charge (pH +4.07), EC (+1.31 dS m^{-1}), OM (+9.80%), CEC [+20.60 cmol(+) kg^{-1}], organic C (+4.42%), total N (+0.27%), available P (+34.50 ppm), and K-exch [+0.41 cmol(+) kg^{-1}] compared with the control. The WDS-derived HS using technical-grade 1 M NaOH is a cost-effective, large-scale organic amendment that improves soil fertility and nutrient availability in highly weathered tropical soils. These findings highlight the potential of utilizing WDS as a circular bioeconomy pathway that converts palm oil processing waste into high-value HS for soil improvement. This strategy simultaneously improves the short-term chemical fertility of acidic Ultisols, enhances nutrient retention and availability, promotes resource efficiency, and reduces reliance on synthetic fertilizers, thus supporting the transition to sustainable and resilient agricultural systems in the tropics.

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