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Extraction and characterisation of humic acid from different organic wastes and its physico-chemical properties

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Abstract

A study on characterization and extraction of humic acid collected from various agro-industrial and agricultural organic wastes from different locations viz., cocopeat, coffee pulp, pressmud, biofuel waste, distillery biocompost, sewage sludge, poultry manure, vermicompost, urban compost and farm yard manure (FYM). Results reveal that physical properties such as bulk density and water holding capacity of different organic wastes ranged from 0.22 Mg m⁻³ (85.50 %) in coco peat to 0.69 Mg m⁻³ (40.21%) of biofuel waste. Chemical properties like pH of poultry manure (pH 8.32) were alkaline in nature and biofuel waste (pH 5.28) slightly acidic in nature. Electrical conductivity was high 1.89 dSm⁻¹ in coffee pulp followed by FYM 1.50 dS m⁻¹. Organic carbon content was higher (40.99%) in biofuel waste. While it was lowest (14.13%) in sewage sludge. With respect to major and secondary nutrients poultry manure recorded higher total N, P, K and Ca (2.85, 1.56, 1.92 and 3.89 %) and iron, manganese, copper and zinc (3850, 541.30, 76 and 476 mg kg⁻¹ respectively). Higher recovery of humic and fulvic acid, (8.92 and 4.21 %, respectively) was recorded in press mud and lowest was in coco peat with 3.43 and 2.45 per cent, respectively. Elemental composition (CHNO) of humic acid extracted from poultry manure had higher N (6.01 %) followed by pressmud and coffee pulp. Higher C (61.04 %) and H (8.89%) were recorded in biofuel waste and O (52.31 %) was higher in sewage sludge. Carboxyl (6.08 meq g⁻¹), phenolic hydroxyl (1.94 meq g⁻¹) groups and total acidity (8.02 meq g⁻¹) was higher in poultry manure followed by pressmud and coffee pulp. However, lower content of carboxyl (2.84 meq g⁻¹) phenolic hydroxyl group (1.32 meq g⁻¹) and total acidity (4.16 meq g⁻¹) was observed in biofuel waste. Slightly higher concentration of nickel (21.8 mg kg⁻¹) and chromium (17 mg kg⁻¹) was observed in humic acid of sewage sludge.

Keywords: Humic acid, fulvic acid, organic wastes, physical and chemical properties, percent recovery, elemental composition

Introduction

Organic matter is considered as the “Life of soil” due to its importance in maintaining fertility of the soil, the depletion of the same will become a major threat to food security in the years to come. Hence, there is a need to improve the soil fertility in a sustainable manner by utilizing the locally available agro-industrial and other organic wastes because these wastes contains substantial amount of nutrients which are necessary for the plant growth in addition to maintaining of soil health. Utilization of these agro industrial wastes and other organic wastes as a source of nutrients reduces the reliance on chemical fertilizers and these organic wastes provides substantial quantity of nutrient elements as well as humus which helps in improving the physical, chemical and biological properties of soil. However, to improve the organic matter content of soils many management techniques have been adopted such as crop rotation, plough techniques, green manuring and application of animal residues, application of humic acids and humates (Doran, 2003)^[2].

Use of bulky organic manures has been considered as a burden by the farmers as it requires large number of labourer for transportation and application. Also, use of bulky organic manures results in spreading of weed seeds in crop land and control of weeds would also be a major problem. In this context, extraction of humic substances from bulky organic manures and their use may help solve many problems associated with use of bulky organic manures.

Many of the agro industrial units viz., sugar mills distilleries etc., generate large quantities of organic wastes which pose problems of disposal and transport. In this context extraction of humic acid from these agro industrial wastes would be a very valuable alternative. The industry would benefit in overcoming disposal and transportation.

Composition of the organic waste varies due to many factors. The composition mainly depends on the type of waste, which is dependent on the raw materials used in the industry or the human population and how often waste is collected and how it is disposed.

Humic acids are heterogeneous, which include macromolecule, hydrophilic acidic functional groups and hydrophobic groups. Humic acid hydrophilic groups promotes hydration and thus increases the water retention capacity in soils. Humic acid (HA) is the main fractions of humic substance (HS) and the most active components of soil and compost. Physically, it promotes good soil structure and

increases the water holding capacity of the soil, biologically it enhances the growth of useful soil organisms, and chemically it serves as an effective adsorption and retention complex for inorganic plant nutrients and there by enhances nutrient uptake and yield by improving the quality and production of crops.

Keeping all these factors in view present investigation entitled "Extraction and characterisation of humic acid from different organic wastes and its physico-chemical properties" was carried out with an objective of Extraction and characterization of humic acid from different organic wastes.

Material and Methods

Characterization of organic wastes

Various agro-industrial and agricultural organic wastes were collected from different locations and the details of collection are furnished in Table 1. The organic waste samples were air dried, powdered and stored for further analysis.

Table 1: Organic wastes from different locations

Humic acid sources	Sources
Coco peat	Cocopeat industry, Doddaballapur
Coffee pulp	Farmers field, Chikkamagalur
Pressmud	Chamundeshwari Distillery, K.M Doddi, Mandya
Biofuel waste	Organic farming unit, UAS, GKVK, Bangalore
Distillery bio compost	Chamundeshwari Distillery, K.M Doddi, Mandya
Sewage sludge	Sewage treatment plant, Jakkur, Bangalore
Poultry manure	Poultry farm, Doddaballapur
Vermicompost	Zonal agricultural research station, UAS, GKVK
Urban compost	Karnataka compost development corporation, Bangalore
Farmyard manure	Farmer's field, Harohalli

Nutrient content of organic waste

Powdered samples of organic material was analyzed for pH and electrical conductivity in 1:10 and 1:100 ratio of organic waste and distilled water. Kjeldhal digestion cum distillation

method was adopted for total N estimation. For other macro, secondary and micro nutrients estimation organic waste samples were digested separately using diacid mixture. The methods adopted for estimation are presented in Table 2.

Table 2: Methods adopted for the analysis of organic wastes

Parameter	Method	Reference
MWHC (%)	Keen's Cup	Piper (1966)
pH	Potentiometry	Jackson (1973)
EC (dS m ⁻¹)	Conductometry	Jackson (1973)
Organic carbon (%)	Dry combustion	Jackson (1973)
Total nitrogen (%)	Micro kjeldahl digestion and distillation	Piper (1966)
Total phosphorus (%)	Vanadomolybdc yellow colour spectrophotometry	Piper (1966)
Total potassium (%)	Flame photometry	Piper (1966)
Total calcium (%)	Versenate titrimetry	Piper (1966)
Total magnesium (%)	Versenate titrimetry	Piper (1966)
Total sulphur (%)	Turbidimetry	Piper (1966)
Total Fe, Mn, Zn and Cu (ppm)	Atomic absorption spectrophotometry	Lindsay and Norvell (1978)
Total B (ppm)	Colorimetry using Azomethane-H reagent with continuous flow analyzer	Page <i>et al.</i> (1982)
Heavy metals content (Pb, Ni, Cr, Cd)	Atomic absorption spectrophotometry	Lindsay and Norvell (1978)

Extraction, fractionation and quantification of humic substances

Extraction of humic substances

Ten gram of air dried organic sample was weighed in to 250 ml conical flask 100 ml of 0.1 N NaOH was added (Schnitzer and Skinner, 1968)^[19] and shaken for 24 hours. Dark colored supernatant solution was separated by centrifugation and collected. The extraction procedure was repeated thrice using 50 ml of extractant each time for complete extraction of the humic substances.

Fractionation and purification of humic substances

Precipitated humic acid fraction was separated by centrifugation. Precipitation and centrifugation was repeated to attain partial purification of humic acid fraction as described by Stevenson (1981)^[20]. The fractions were further purified by treating with HCl-HF mixture (5 ml of each HCl and HF acids were dissolved in 990 ml of double distilled water) for 24 hours and this acid mixture was separated by centrifugation. The residue so obtained was thoroughly washed with distilled water and freeze dried to obtain humic acid.

higher organic carbon content as reported by Francou *et al.* (2005)^[3].

The organic carbon content was higher (40.99%) in biofuel waste while it was lowest (14.13%) in sewage sludge. With respect to major nutrients poultry manure recorded higher total N, P, K and Ca (2.85, 1.56, 1.92 and 3.89 %, respectively) followed by higher N (2.01 %), in coffee pulp and P (1.27 %) in pressmud. Similar findings were recorded by Mohamed *et al.* (2010). However lowest N (0.42 %) was in FYM, P content was low (0.12%) in coco peat and sewage sludge recorded low K (0.2 %). Among secondary nutrients higher calcium content was recorded in poultry manure (3.89 %). With respect to magnesium and sulphur higher (5.84 and 0.75%) was recorded in sewage sludge. Low magnesium (0.86 %) and sulphur (0.24 %) was in FYM. This might be due to variations in composition of different waste materials used for decomposition. Higher content of sulphur in sewage sludge can be attributed to presence of chemical alkyl benzene sulfonate which is a widely surfactant in detergent formulations which is a common commodity used in households (Smith, 2009). higher concentrations 3850, 541.30, 76 and 476 mg kg⁻¹ of iron, manganese, copper and zinc, respectively was observed in poultry manure followed

by higher iron and manganese content in pressmud (3642 and 525 mg kg⁻¹, respectively) and coffee pulp (3407 and 480.7 mg kg⁻¹, respectively). However, higher copper (93 mg kg⁻¹) and zinc (370 mg kg⁻¹) was in sewage sludge. Lower concentration of 1602, 182.20, 27.20 and 25.10 mg kg⁻¹ of iron, manganese, copper and zinc was in biofuel waste. The sewage sludge recorded higher boron content (85.40 mg kg⁻¹) followed by poultry manure (72.28 mg kg⁻¹) and it was lower (25.87 mg kg⁻¹) in biofuel waste. Similar results were obtained by Gomez *et al.* (1993)^[6] who reported that the nutrient contents of different organic materials act as an alternative nutrient source with beneficial effect in improving soil properties and supply of nutrients for adequate plant growth and development.

Among heavy metals in organic wastes higher (41.7 mg kg⁻¹) of nickel was observed in urban waste while cadmium (8.75 mg kg⁻¹), lead (36.1 mg kg⁻¹) and chromium (33.91 mg kg⁻¹) were higher in sewage sludge. Lead and cadmium were not detected in other organic samples except sewage sludge and urban compost. The results are in agreement with studies conducted by Manju *et al.* (2013)^[12] on characterization of MSW and also by (Gomez *et al.*, 1993)^[6].

Table 3: Physico-chemical properties, major and secondary nutrient composition of different organic wastes used for extracting humic acid

Organic wastes	BD (Mg m ⁻³)	WHC (%)	pH 1:50	EC (dSm ⁻¹) 1:100	OC (%)	Major nutrients			Secondary nutrients		
						N	P	K	Ca	Mg	S
Coco peat	0.22	85.50	5.68	1.19	35.61	0.43	0.12	0.49	1.64	0.79	0.26
Coffee pulp	0.53	40.86	7.78	1.49	38.36	2.01	0.52	1.56	3.44	1.05	0.41
Pressmud	0.54	41.78	7.18	1.12	36.11	1.75	1.27	1.25	3.72	1.15	0.46
Biofuel waste	0.69	40.21	5.28	1.05	40.99	0.61	0.16	0.47	2.00	0.85	0.20
Distillery biocompost	0.51	47.42	8.03	1.59	18.56	1.59	1.04	1.40	3.20	1.42	0.31
Sewage sludge	0.55	42.50	5.95	1.12	14.13	0.43	1.03	0.20	3.54	5.84	0.75
Poultry manure	0.52	48.05	8.32	1.26	34.29	2.85	1.56	1.92	3.89	1.93	0.48
Vermicompost	0.58	44.00	6.52	0.99	18.26	0.74	0.30	0.47	1.76	0.89	0.28
Urban compost	0.56	45.23	7.59	1.36	24.01	1.24	0.49	1.15	3.08	1.62	0.42
FYM	0.49	43.29	7.12	1.50	17.43	0.42	0.35	0.51	1.52	0.86	0.24

Table 4: Micronutrients and heavy metals content of different organic wastes used for extracting humic acid

Organic wastes	Micronutrients					Heavy metals				
	Fe	Mn	Cu	Zn	B	Ni	Cd	Pb	Cr	
	(mg kg ⁻¹)									
Coco peat	2049	309.20	29.40	32.30	45.20	29.98	BDL	BDL	24.20	
Coffee pulp	3407	480.70	63.50	225.00	56.80	30.19	BDL	BDL	25.03	
Pressmud	3642	525.00	67.50	182.40	64.20	33.80	BDL	BDL	23.57	
Biofuel waste	1602	182.20	27.20	25.10	25.87	26.09	BDL	BDL	20.41	
Distillery biocompost	2091	312.00	66.90	101.00	59.15	29.10	BDL	BDL	25.90	
Sewage sludge	3450	365.60	93.00	370.60	85.40	31.24	8.75	36.1	33.91	
Poultry manure	3850	541.30	76.00	476.00	72.28	27.87	BDL	BDL	19.23	
Vermicompost	2897	282.30	48.00	83.60	51.65	34.48	BDL	BDL	26.29	
Urban compost	3025	319.10	71.20	112.00	69.48	41.70	BDL	14.6	20.26	
FYM	1968	264.40	41.90	85.00	43.27	30.24	BDL	BDL	23.45	

Note: BDL – Below detectable limit

Percent recovery of humic acid and fulvic acid from different organic wastes

The results of the recovery percentage of humic acid and fulvic acid from different organic wastes are presented in Table 5. Higher recovery of 8.92 and 4.21 per cent humic and fulvic acid was recorded for press mud followed by coffee pulp and poultry manure. The results are in concurrence with work carried out by Satisha and Devarajan *et al.* (2011)^[16]

and Gayathri *et al.* (2011)^[5]. However lower humic and fulvic acid 3.43 and 2.45 per cent was recorded in coco peat, respectively. Scheild *et al.* (1989) reported low recovery of humic acid in sewage sludge materials. The humic acid extracted from different organic wastes is dark brown to black in color, which might be due to elemental configuration and melanin pigment (Kumuda, 1987)^[11].

Table 5: Recovery percentage of humic and fulvic acid from different organic wastes

Organic wastes	Humic acid (%)	Fulvic acid (%)
Coco peat	3.43	2.45
Coffee pulp	7.42	3.21
Pressmud	8.92	3.59
Biofuel waste	6.09	2.59
Distillery biocompost	3.62	2.57
Sewage sludge	4.87	2.75
Poultry manure	8.20	3.88
Vermicompost	4.98	3.76
Urban compost	6.30	2.85
FYM	4.17	3.17

Elemental composition and molar ratios of humic acid extracted from different organic wastes

The elemental composition and molar ratios of humic acid extracted from different organic wastes are presented in Table 6. The humic acid extracted from biofuel waste recorded higher content of carbon (61.04 %) followed by coco peat (50.91 %) and distillery biocompost (48.48 %) followed by coffee pulp and pressmud (47.10 and 46.4 %, respectively). Sathisha and Devarajan (2011)^[16] also reported 46.36 per cent of organic carbon in the humic acid extracted from pressmud which may be due to higher organic carbon content of the test materials in present study. With respect to hydrogen, higher (8.89 %) concentration was recorded in biofuel waste sample and lower concentration (4.08 %) was observed in poultry manure. The nitrogen content of humic acid was reflected by nature of raw material. Poultry manure recorded higher (6.01 %) nitrogen followed by pressmud (5.37 %), coffee pulp (5.07 %) and urban compost (4.95 %) while least was in FYM (2.32 %). Similar findings were observed with respect to urban compost and sewage sludge (Atiyeh *et al.*, 2002 and Keiji Jindo *et al.*, 2012)^[1,9]. With respect to oxygen content, higher value (52.31 %) was recorded in sewage sludge followed by FYM (52.19 %) and vermi compost (47.56 %).

The results of the molar ratios of elements suggest the stoichiometric relationship that exist among the elements. The H/C ratio in different organic samples ranged from 0.09 to 0.15. Lower H/C ratio in poultry manure (0.09) followed by coffee pulp, press mud and urban compost suggest that polymerisation and or condensation takes place well, due to introduction of carbohydrate and oxidation of phenolic compounds with methoxyl groups or aliphatic side chain in the humic acids (Vila *et al.*, 1982)^[22]. With respect to O/C ratio higher (1.30) was recorded in sewage sludge and FYM, and lower (0.44) was recorded in biofuel waste. The presence of C, H and O in humic acid is accounted in the form of carboxyl, methoxyl and carbonyl groups (Govindasamy *et al.*, 1989)^[7]. These values are highly helpful in judging the extent of the role dehydrogenation as well as removal of alkyl or carboxyl groups during the operational process of extracting humic acid. The difference in the ratios studied could be attributed to the dissimilar splitting of the peripheral aliphatic chains as well as to an increase in the number and length of aliphatic chains (Govindasamy *et al.*, 1989; Kononova, 1966)^[7, 10]. The N/C ratio was higher in poultry manure (0.13) followed by press mud (0.12), coffee pulp (0.11) and urban compost (0.11) indicating that acid insoluble humic nitrogen increased considerably.

Table 6: Elemental composition and molar ratios of humic acid extracted from different organic wastes

Organic wastes	Contents of elements (%)				Molar ratios of elements		
	C	H	N	O	H/C	N/C	O/C
Coco peat	50.91	5.80	3.05	40.24	0.11	0.06	0.79
Coffee pulp	47.10	4.40	5.07	43.43	0.09	0.11	0.92
Press mud	46.40	4.35	5.37	43.88	0.09	0.12	0.95
Biofuel waste	61.04	8.89	3.01	27.06	0.15	0.05	0.44
Distillery biocompost	48.48	4.91	4.09	42.52	0.10	0.08	0.88
Sewage sludge	40.35	4.39	2.95	52.31	0.11	0.07	1.30
Poultry manure	45.06	4.08	6.01	44.85	0.09	0.13	0.99
Vermicompost	44.54	5.06	2.84	47.56	0.11	0.06	1.07
Urban compost	46.57	4.39	4.95	44.09	0.09	0.11	0.95
FYM	40.02	5.47	2.32	52.19	0.14	0.06	1.30

Functional groups of humic acid extracted from different organic wastes

The data on functional groups and E₄/E₆ ratio of humic acid extracted from different organic wastes are presented in Table 7. Higher content of carboxyl group (6.08 meq g⁻¹), phenolic hydroxyl group (1.94 meq g⁻¹) and total acidity (8.02 meq g⁻¹) was recorded in poultry manure, this was followed by pressmud and coffee pulp. While lower content of carboxyl (2.84 meq g⁻¹), phenolic hydroxyl group (1.32 meq g⁻¹) and total acidity (4.16 meq g⁻¹) was observed in biofuel waste. Higher content of carboxyl group compared to phenolic hydroxyl group suggest that the carbohydrates and phenolic compounds present in these substances are easily degradable

and are readily converted to carboxyl group on subsequent oxidation (Masaaki *et al.*, 1992)^[13]. Similar results were reported by Satisha and Devarajan (2011)^[16]; Ushashree *et al.* (1989)^[21]. The higher acidity or exchange-capacity of these humic substances could be attributed to the occurrence of ionisable H⁺ ions of carboxyl and hydroxyl groups found in aliphatic chains or aromatic rings of molecules (Schnitzer, 2000). It is also evident from the present study that carboxylic and phenolic hydroxyl groups were present in varying quantities and these findings corroborate with that that of Prasad and Sinha (1981)^[15] who reported that the variation in molecular weight may be responsible for the difference in the quantities of functional groups.

The E_4/E_6 ratio is a valid and informative index for characterization of humic substance with respect of aromaticity (Kononova, 1966) [10]. In the present study, the E_4/E_6 ratio was high in humic acid extracted from poultry manure (5.21) followed by press mud (5.12), coffee pulp (5.10) and urban compost (5.01) lower E_4/E_6 ratio was

recorded in biofuel waste (4.58). Higher E_4/E_6 ratio indicates more aliphatic nature of the fractions (Garcia *et al.*, 1991) [4] and in turn also reflects a low degree of aromatic condensation. Similar observations were also reported by Kadalli *et al.* (2000) [8].

Table 7: Functional groups and E_4/E_6 ratio of humic acid extracted from different organic wastes

Organic wastes	Total acidity (meq g ⁻¹)	Carboxylic group (COOH) (meq g ⁻¹)	-OH (Phenolic groups) (meq g ⁻¹)	E_4/E_6 ratio
Coco peat	5.03	3.60	1.43	4.70
Coffee pulp	7.68	5.89	1.79	5.10
Pressmud	7.84	5.97	1.87	5.12
Biofuel waste	4.16	2.84	1.32	4.58
Distillery biocompost	5.57	3.79	1.58	4.95
Sewage sludge	5.40	3.73	1.47	4.78
Poultry manure	8.02	6.08	1.94	5.21
Vermicompost	5.12	3.78	1.34	4.85
Urban compost	7.41	5.75	1.71	5.01
FYM	4.92	3.53	1.39	4.61

Summary

Present study revealed that utilization of agro industrial wastes and other organic wastes as a source of nutrients reduces the reliance on chemical fertilizers and these organic wastes provides substantial quantity of nutrient elements as well as humus which helps in improving the physical, chemical and biological properties of soil. Intern soil health by decreasing demand on chemical fertilizers and also reduce soil pollution, Thus, humic acid derived from organic wastes like cocopeat, pressmud, coffee pulp, sewage sludge, poultry manure, urban compost etc. which have substantial quantities of humic materials are of great importance in maintaining soil organic matter levels especially in semi-arid tropics of India.

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