

Characteristics of humic acids of Mukah coal in Sarawak

(Sifat asid humik arang batu dari Mukah, Sarawak)

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Key words: Mukah, Sarawak, Malaysian coal, humic acids, nitrohumic acids, regenerated coal

Abstract

In Malaysia, abundant coal resources were found in Sarawak and Sabah. The utilization of coal resources, to date, is emphasized on the energy productions. The non-energy utilization as soil conditioner is unexplored. Therefore, this study attempted to characterize the coal humic acids extracted from Mukah coal and to evaluate its properties as soil conditioner. The coal humic acids from the regenerated sample were also assessed.

The results revealed that different extractants, concentrations, mesh size and temperature influenced the properties of humic acids. The extraction with KOH at 0.5 M produced humic acids with low ash content and high acidic functional groups, which are substantial as soil conditioner. However, the essential yield was low (20%). Regeneration of coal sample with 10% nitric acids improved the yield to an average of 83.45%. The acidic functional groups of nitrohumic acids were improved with the ash content remained at a low level. The UV-VIS study revealed no significant difference on the humification degree of humic acids prepared with varying treatments. Nevertheless, the nitration process was found to result in humic acids with higher condensation level.

Introduction

Humic acids are found in various environments such as soils, natural waters, rivers, lakes, sea sediment, plants, peat and composts. It has been widely used to condition physical, chemical as well as biological properties of soil to improve agricultural productivity. Addition of humic substances extracted from an oxidized coal to soil was found to retard the onset of runoff process under rainfall (Piccolo and Celano 1994). Furthermore, an increase of soil cation exchange capacity, pH and

organic matter was observed after incorporation of coal derived humic acids on two Nigerian nutrient poor tropical soils (Piccolo et al. 1997).

A number of natural sources can be considered as raw material for the manufacture of humic acids based commercial specialties. Nevertheless, coal appears to be one of the potential sources with high humic acids content. The low-grade coal recorded 40–85% of humic substances compared to black peat, 10–40%; sapropel peat, 10–20%; brown coal, 10–30%;

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compost, 2–5%; soil and sludge, 1–5%. Coal resources in Malaysia to date are estimated at 1,050 million tonnes. Coal resources are mainly found in Sarawak and Sabah with 69% and 29%, respectively (Thaddeus 2000). The quality of the coal resources may range from lignite to anthracite; bituminous to sub-bituminous.

To date, the utilization of coal resources is specifically for energy generation. An estimated 300,000 tonnes of coal from Merit-Pila Field (Kapit, Sarawak) was produced for Sejingkat Power Plant in Sarawak in 1999. The non-energy utilization of the coal resources as soil conditioner remains unexplored. Therefore, the objectives of this study were to extract and to characterize humic acids derived from the indigenous coal, and to assess the suitability of the extracted humic acids as soil conditioner.

Materials and methods

Extraction of humic acids

Alkali extraction The extraction method described by the International Humic Substances Society (1983) was adopted with modifications. Mukah coal sample was pulverized, washed and sieved through 50 μm sieve to eliminate the coal dust. The retained coal sample was oven-dried at 105 °C for 24 h. Coal sample of 10 g weight was added to 100 ml of NaOH (0.1 M) and was heated to 70 or 100 °C for 2 h.

The supernatant was filtered through 50 μm sieve and the coal residue was washed with distilled water until the washed water was clear. The coal residue was dried at 105 °C and weighed. The supernatant was acidified with concentrated H_2SO_4 to pH 1–2 and allowed to stand for 24 h. The precipitated humic acids were separated by centrifugation at 6,000 rpm for 10 min. The humic acids were washed with distilled water and centrifuged twice. The gel-like humic acids were oven-dried at 60 °C and stored in desiccators. The procedure of extraction was repeated with 0.5 M and 1.0 M of NaOH and 0.1 M, 0.5 M and

1.0 M of KOH. To investigate the effect of mesh size and temperature, the washed coal was sieved using 710 μm , 2 mm and 4 mm mesh size and extracted at temperatures of 70 °C and 100 °C.

Nitration of coal The coal sample was oxidized to produce regenerated coal prior to extraction in an attempt to improve the yield of humic acids. The regenerated coal was obtained by nitrating the coal sample with nitric acids at 5, 10 and 20% (1 g of coal with 5 ml of nitric acids) at 70 °C for 2 h with continuous stirring. The coal sample was dried in oven at 60 °C. The nitrated coal was subjected to the alkaline extraction and the resulting extracts were referred to as nitrohumic acids.

Yields

The yield of humic acids was calculated as the weight of extracted humic acids per unit weight of coal.

Characterization of humic acids

Moisture The moisture content was determined by drying the samples at 105 °C overnight. The ash content was determined by combustion of samples at 800 °C for 2 h (Allen 1989).

Total acidity, carboxylic and phenolic content The total acidity was measured using the barium hydroxide method and the carboxyl groups were determined using calcium acetate method. The phenolic content was calculated as the difference between the total acidity and the carboxyl groups (Schnitzer 1977).

Total nitrogen content The nitrogen content was analysed with Kjeldahl digestion procedure (Chin 2000).

FTIR analysis FTIR spectra of the samples were recorded on KBr pellets (2 mg humic acids and 100 mg KBr) using a Shimadzu Series FTIR spectrophotometer.

All of the spectra were self-deconvoluted to improve the apparent resolution.

UV-VIS analysis Approximately 5 mg of humic acid sample was dissolved in 25 ml of 0.05 M NaHCO₃ solutions with pH adjusted to 8–9 with 0.1 M NaOH to assist solubility. Absorbances at 400, 465, 600 and 665 nm were recorded. The E₄/E₆ value was calculated based on the ratio of absorbance at 465 nm and 665 nm. Absorbance at 465 nm expresses presence of humic acids formed in initial humification stage and absorbance at 665 nm indicates presence of humic acids formed in well-humified organic matter (Debska et al. 2002). The value of $\Delta \log K$ coefficient, $\Delta \log K = \log A_{400} - \log A_{600}$, was calculated to categorize the degree of humification (Kumada 1975).

Statistical analysis

Statistical analysis was performed with single-factor ANOVA to identify the significant difference in yield of humic acids due to different extraction conditions i.e. temperature and mesh size. Analysis was also executed to evaluate the humification degree of humic acids extracted.

Results and discussion

Direct alkaline extraction

Humic acids were extracted from the indigenous coal with different extractant and varying conditions (concentration, mesh size and temperature). *Table 1* summarizes the extraction yield with NaOH and KOH at different concentrations. Generally, the yield of humic acids was increased with the

Table 1. Yield of humic acids under different extraction conditions

Extraction conditions	Concentration (M)	Yield (%)
NaOH	0.1	3.61
	0.5	9.83
	1.0	13.67
KOH	0.1	3.43
	0.5	16.00
	1.0	20.00

concentration of alkaline solution. Yates and Von Wandruszka (1999) reported an increase in surface tension with concentration of Na⁺, which indicated that humic solute was more anionic and therefore hydrophilic at higher molarity. The KOH extraction was found to be more efficient than NaOH extraction. The extraction performance of KOH has been well documented. Garcia et al. (1996) reported the highest extraction efficiency with 0.25 M KOH compared to NaOH and Na₄P₂O₇. The observation was supported by Rocha et al. (1998). KOH possesses a greater efficiency, as the ionic radius of K⁺ (300 x 10⁻⁹m) is smaller in relation to Na⁺ (450 x 10⁻⁹m). This allows a greater expansion, counterbalance and capacity of interaction of the intra and intermolecular charges present in the humic substances (Rocha et al. 1998). Generally, the maximum extraction yield was obtained at an average of 20% with KOH. Re-extraction of humic acids produced an average yield of <2% from 0.5 and 1.0 M of KOH which did not reflect a significant improvement on the yield. However, manipulating the mesh size of coal and the temperature of extraction has improved the yield.

Figures 1–2 show the influence of mesh size and temperature on the extraction yield. Finer grains of coal and higher temperature could possibly increase the extraction yield marginally although statistical analysis at 95% confidence level revealed no significant difference in the yield of humic acids at varying temperatures and mesh sizes.

The essential yield of humic acids is economically unviable to be used as soil conditioner. In comparison to leornadite marketed elsewhere (contains average of 80% of humic acids), the humic acids extracted from the indigenous coal were far too little for economically sustainable exploitation.

Nitrohumic acids extraction

Pretreatment of the coal samples with nitric acids (HNO₃) has increased the yield

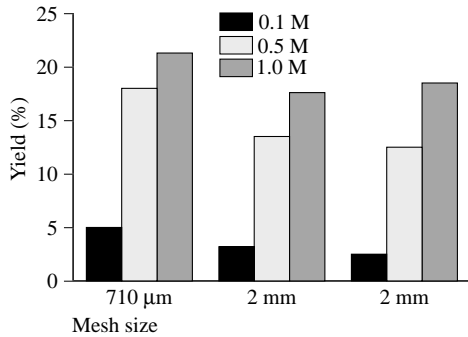


Figure 1. Influence of mesh size on the extraction yield

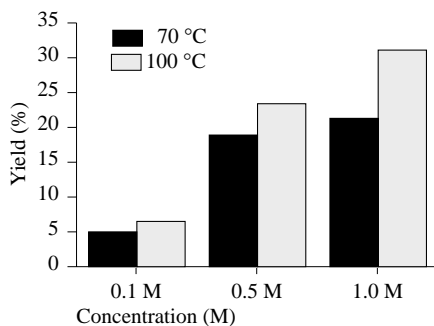


Figure 2. Influence of temperature on the extraction yield

Table 2. Yield of humic acids with pretreatment of nitric acids

Pretreatment condition	Yield (%)
5% HNO ₃	67.92
10% HNO ₃	83.45
20% HNO ₃	65.12

substantially. The role of HNO₃ in regeneration process was to manipulate the solubility of humic acids by introducing acid groups from HNO₃ to enable them more readily attacked during extraction.

Therefore, the yield of humic acids is governed by the concentration of HNO₃ in the nitration process. Table 2 summarizes the yield of humic acids with pretreatment of nitric acids prior to extraction with 0.5 M KOH. Pretreatment of the coal samples with 10% HNO₃ improved the yield tremendously to an average of 83.45%. Improvement as such was also reported in the study by Won (1968) as the yield of nitrohumic acids is five times (by weight) more than the original humic acids.

Chemical characteristics of humic acids

Chemical characteristics of humic acids are one of the determinants for the effectiveness of the products as soil conditioner. Table 3 presents the chemical characteristics of humic acids extracted with NaOH and KOH at varying concentrations. The moisture content of the humic acids was the result of sample preparation. The ash content, however, may be due to the sample preparation or the origin of raw materials. An average of 16.96% ash in the humic acids extract would likely attribute to the residual Na₂SO₄/K₂SO₄ from preparation process as the value exceeded the ash content of the raw material (0.78%).

Table 3. Characteristics of humic acids with varying extractants and concentrations

Extraction condition	Moisture (%)	Ash (%)	Functional groups (meq/g)			Nitrogen (%)
			Total acidity	-COOH	-OH	
0.1 M NaOH	4.44 ± 1.93	25.56 ± 1.93	6.36 ± 0.04	2.42 ± 0.03	3.95 ± 0.06	0.68 ± 0.04
0.5 M NaOH	18.59 ± 2.37	14.10 ± 0.17	8.17 ± 0.05	2.10 ± 0.06	6.07 ± 0.10	0.68 ± 0.03
1.0 M NaOH	29.04 ± 4.73	16.83 ± 0.80	7.93 ± 0.05	1.52 ± 0.03	6.41 ± 0.05	0.55 ± 0.04
0.1 M KOH	11.68 ± 3.07	20.39 ± 0.34	7.17 ± 0.03	1.60 ± 0.04	5.57 ± 0.06	0.65 ± 0.04
0.5 M KOH	8.09 ± 1.73	10.06 ± 0.92	11.49 ± 0.04	2.27 ± 0.05	9.22 ± 0.05	0.65 ± 0.03
1.0 M KOH	9.89 ± 3.34	23.08 ± 7.81	8.36 ± 0.02	2.76 ± 0.03	5.60 ± 0.03	0.43 ± 0.05
10% NO ₃ /0.5 M KOH	5.42 ± 1.82	8.68 ± 1.77	11.28 ± 0.04	3.59 ± 0.05	7.69 ± 0.04	1.65 ± 0.05

The ash content indicated a decrease at 0.5 M as compared to 0.1 M and 1.0 M for both extractants. The observation was not explainable nevertheless; similar observation was recorded in the study of Rosa et al. (2000). Levesque and Schnitzer (1966) in addition revealed a reduction of ash content from 25% to 5% with 0.4 N NaOH. Humic acids with high ash content may not serve as an effective soil conditioner as the concentration of humic acids is the key distinguishing factor for its effectiveness in the field.

Considering the total acidity, humic acids extracted with KOH generally consisted more acidic functional groups compared to NaOH. According to Piccolo et al. (1992), the most effective humic fractions were those with higher acidic functionality. The extraction with KOH could probably produce a better quality humic acids product. The promoted auto-oxidation process with KOH extraction has resulted in higher C=O content. The nitration process further improved the total acidity properties of humic acids, enhancing its feature as soil conditioner. During nitration process, the core structure of humic acids are degraded into a variety of aliphatic dicarboxylic acids, benzenecarboxylic acids,

hydroxybenzoic acids and nitro compounds such as nitrophenols, nitrobenzoic acids and dinitrosalicylic acids (Stevenson 1982). The formation of these compounds was reflected in the increase of carboxyl and phenolic content. The oxidation process in addition substitutes the nitro and nitroso groups to phenolic structures of humic acids which was evidenced in the increase of nitrogen content (Gavilan et al. 1982; Patti et al. 1996).

FTIR

There are several important features on the IR spectra of humic acids extracted with KOH and NaOH (*Figures 3–4*).

- A weak sharp band at 3,690/cm which is attributed to the valence vibration of water
- A broad band around 3,400/cm due to O–H stretching of phenol and alcohol
- A couple of weak bands between 2,920 and 2,850/cm attributed to C-H aliphatic
- A well-defined band at 1,706 /cm due to C=O stretching of ketonic and carboxylic groups
- A strong peak at 1,620/cm assigned to aromatic C=C stretching, C=O of conjugated ketones and carboxylate ions

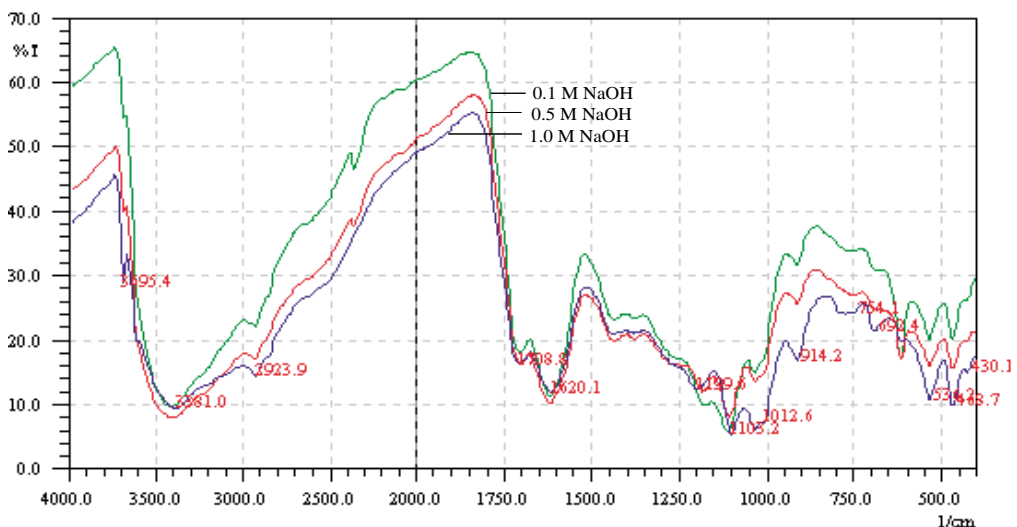


Figure 3. FTIR spectra of humic acids extracted with different concentrations of NaOH

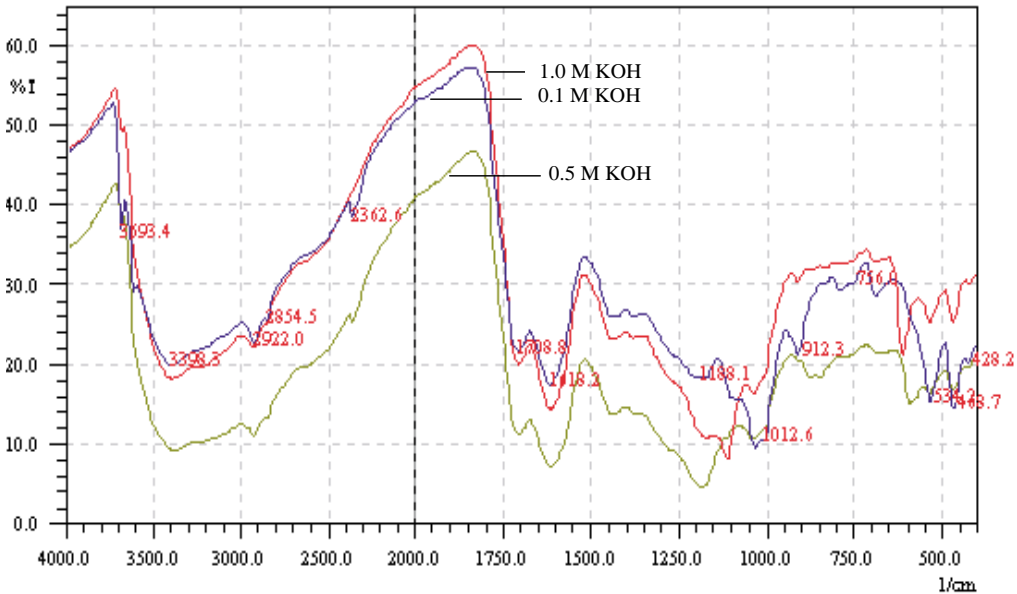


Figure 4. FTIR spectra of humic acids extracted with different concentrations of KOH

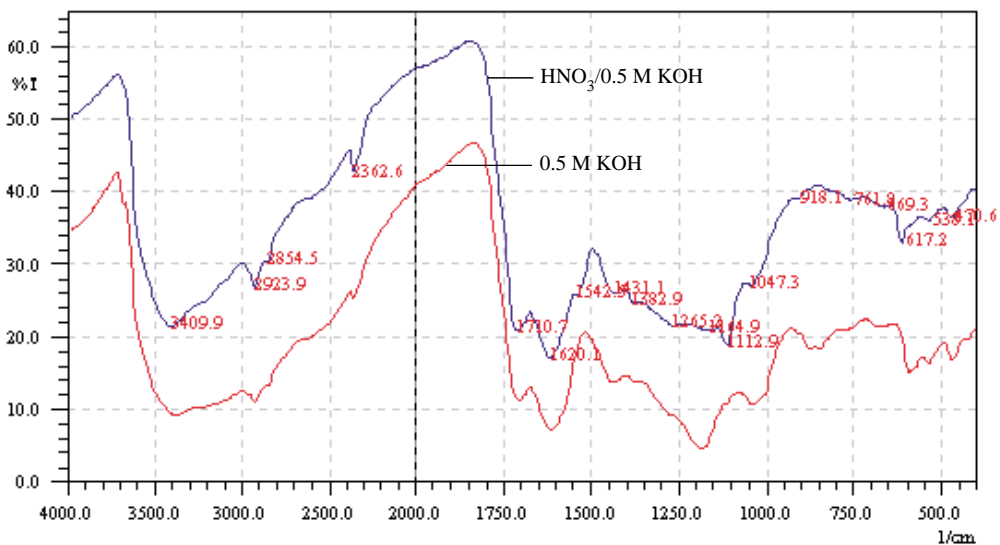


Figure 5. FTIR spectra of humic acids prepared from nitrated coal and untreated coal with 0.5 M KOH

- A couple of peaks at 1,430/cm and 1,334/cm due to C–H stretching
- A group of low intensity bands between 1,000/cm and 1,100/cm attributed to Si–O stretching and C–O stretching of polysaccharides
- Weak bands at 1,040/cm due to S=O due to treatment of humic acids with sulphuric acids
- Absorption bands at 470–480/cm and 530–540/cm which are due to mineral components.

The spectra of the isolated humic acids were similar to the others reported elsewhere (Rocha et al. 1998; Novak et al. 2001; Dick et al. 2002). However, there are relatively more bands in the region of 400–700/cm.

Humic acids may differ significantly with respect to their contents of inorganic constituents, which manifest in the region of 470–480 and 530–540/cm. The variation may be due to the differences in the extraction and purification procedures. Usually treatment with HF/HCl is performed to eliminate the ash content in the humic acids extraction. In the present study, the purification process was excluded therefore the impurities due to mineral components are expected.

The spectra of humic acids prepared with NaOH and KOH exhibited similar pattern. The humic acids extracted with varying molarity of NaOH demonstrated closer similarity compared to KOH extracts, suggesting an interrelated structure. The spectra of humic acids prepared with 0.1 M and 1.0 M of KOH showed a decrease in band intensity at 3,400/cm, 1,700/cm and 1,620/cm. The reduction in the intensity at 1,700/cm may be attributed to the substitution of carbonyl groups to salts as a result of higher ash content in the extracts. The absorption bands presented in the region of 1,000–1,100/cm were shifted for humic acids extracted with varying concentrations of KOH. In this region, absorption bands are assigned to the presence of mineral content where the identification is complicated.

Figure 5 shows the IR spectra of humic acids prepared from the regenerated coal and the untreated coal with 0.5 M KOH. Several changes due to regeneration were observed on the IR spectra: a) an additional poorly-defined shoulder at 1,544/cm attributed to amide II (possibly nitrogroups in humic acids oxidized by nitric acids); b) absence of a band at 1,200/cm with formation of a band at 1,110/cm; and c) increase in band intensity at 1,040/cm. The absence of band at 1,200/cm (carboxylic groups) with the increase of band at 1,040/cm (C–N bonding) suggested that the nitration might occur at the expenses of the carboxylic groups. These observations were slightly deviated from the study of Dick et al. (2002). A sharp absorption band at 1,530/cm attributed to

NO₂ stretching, which was reported by the author, was not noticed in the present study.

UV-VIS

The E₄/E₆ coefficient is widely used in agricultural practices to express the degree of humification. A low ratio may be indicative of a relatively high degree of condensation of aromatic constituents; a high ratio infers the presence of relatively more aliphatic structures (Stevenson 1982). Piccolo et al. (1992) regarded that the aliphatic and aromatic content of the humic acids extract did not play a role in its effectiveness as soil conditioner. Despite that, it is a useful indicator for the molecular size. The E₄/E₆ ratio decreases with increasing molecular weight. Humic fractions with smaller molecular size are anticipated to perform more effectively in regulating the plant growth and nitrate uptake.

Table 4 shows the UV-VIS spectral properties of humic acids. The E₄/E₆ ratios progresses in an increasing trend, although variably, when alkaline media with higher molarity was applied. The results demonstrated that there was a possible structure fractionation with higher concentration of alkaline media leading to a smaller molecular fraction. The observation was likewise reported by Rosa et al. (2000) and Levesque and Schnitzer (1966). In comparison to the extractant employed, the E₄/E₆ of humic acids yielded with KOH were relatively higher compared to those extracted with NaOH, indicative of more aliphatic structure and smaller molecular fractions. This finding supported the results of carboxyl and phenolic content that chemical changes were more drastic with KOH extraction. Nevertheless, the extracts possess an enhanced feature as soil conditioner. The nitration process on the other hand, produced humic acids with lower E₄/E₆ ratio demonstrating higher level of condensation and greater molecular weight. It was well recognized that the degradation products of oxidation process

Table 4. UV-VIS spectral properties of humic acids

Extraction conditions	Concentration (M)	E_4/E_6	$\Delta \log K$
NaOH	0.1	3.372	0.555
	0.5	3.765	0.572
	1.0	3.709	0.590
KOH	0.1	3.642	0.579
	0.5	3.530	0.566
	1.0	4.206	0.642
Regenerated with 10% HNO_3 extracted with KOH	0.5	3.334	0.660

were enriched with aromatic compounds (Orlov 1985).

Kumada (1975) correlated the values of $\Delta \log K$ coefficient and the degree of humification. On the basis of $\Delta \log K$ coefficient, humic acids were divided into three types: type A = humic acids of high degree of humification for which the values of $\Delta \log K$ coefficient are up to 0.6, type B = those of corresponding values between 0.6–0.8, and type $R_p = \Delta \log K$ coefficient within the range of 0.8–1.1. In agricultural perspective, fertilizer with higher humification degree is expected to have a better agronomic quality. The results of $\Delta \log K$ coefficient indicated no significant difference at 95% confidence level for the humic acids prepared with varying treatments.

Conclusion

The study concluded that the extraction yields were dependent on the extractant, concentration, mesh size and temperature. KOH extraction produced a better yield compared to NaOH. The extraction with KOH at 0.5 M generated humic acids with higher acidic functional groups and lower ash content. However, the yield was economically inadequate therefore constraining the application of coal humic acids as soil conditioner. The nitration process enhanced the features of humic acids as soil conditioner as the yield and the acidic functional groups were improved significantly with the ash content remained

low. The IR spectra of humic acids extracted with KOH and NaOH were similar. The IR spectrum of nitrohumic acids, however, revealed an additional poorly defined shoulder at 1,544/cm and the disappearance of the band at 1,200/cm with increased band intensity at 1,040/cm as an indication of nitrogen incorporation. The UV-VIS spectral properties of humic acids did not demonstrate a significant difference to enable this parameter dependable as indicator of humification degree.

Acknowledgement

The authors wish to thank the IRPA grant (01-03-03-0107EA001) and the Universiti Malaysia Sarawak for financial support.

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Abstrak

Di Malaysia, banyak sumber arang batu telah dijumpai di Sarawak dan Sabah. Pada masa ini, ia digunakan untuk tujuan penjanaan tenaga. Penggunaannya sebagai perawat tanah dalam bidang pertanian masih belum diselidiki. Dengan demikian, kajian ini dilakukan untuk memahami sifat asid humik yang diekstrak daripada arang batu Mukah dan potensinya sebagai perawat tanah. Kesan proses regenerasi terhadap sifat asid humik juga dikaji.

Keputusan kajian menunjukkan bahawa jenis pengekstrak, kepekatan, saiz zarah arang batu dan suhu mempengaruhi sifat asid humik terhasil. Pengekstrakan dengan 0.5 M KOH menghasilkan asid humik dengan kandungan abu yang rendah dan kandungan kumpulan berfungsi berasid yang tinggi. Walau bagaimanapun, hasil asid humik yang diperolehi adalah rendah (dalam lingkungan 20%). Proses regenerasi dengan 10% HNO₃ meningkatkan hasil asid humik kepada 83.45% dengan kandungan abu yang rendah dan kandungan kumpulan berfungsi berasid yang tinggi. Kajian UV-VIS tidak menunjukkan perbezaan yang nyata dalam tahap humifikasi asid humik yang disediakan melalui pelbagai rawatan. Walau bagaimanapun, proses penitratiran menghasilkan asid humik yang mempunyai tahap kondensasi yang lebih tinggi.