ISSN: 2277-9655
Impact Factor: 5.164
CODEN: IJESS7

IJESRT
INTERNATIONAL JOURNAL OF ENGINEERING SCIENCES & RESEARCH TECHNOLOGY

ASSESSMENT OF TRANSPORT MATERIAL MAINLAND BASED ON THE COMPOSITION OF LIGNIN PHENOLS IN COASTAL WATER OF THE SPERMONDE ARCHIPELAGO, INDONESIA

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DOI: 10.5281/zenodo.1252818

ABSTRACT

Measurements of lignin-phenols monomers from mainland effluents are used to tracing riverine inputs and diagenesis processes of terrigenous dissolved organic carbon (DOC) to the riverine. The purpose of this study is to analyze the transport of mainland material on the west coast of South Sulawesi based on lignin phenols monomer composition, and bulk elemental (rasio C/N and stable isotope carbon values, $\delta^{13}C$) on dissolved organic carbon material. Sea water samples were collected in October 2017 (dry season) and March 2018 (rainy season), from coastal waters of Tallo-Makassar river estuary and Pangkep. The monomer composition of lignin phenols (Λ) in the rainy season is higher (2.15-3.14) than in the dry season (1.02-1.63). Λ values (the sum of six lignin phenols, expresses as mg/100 mg organic carbon (OC) ranged from 0.050 to 0.142 in the dry season, and 0.417 to 0.491. Ratios of syringyl/vanillyl (S/V) and cinnamyl/vanillyl (C/V) ranged from 0.02 to 1.95 and 0.02 to 4.32, respectively. It is indicating the presence of non-woody angiosperm tissues. The high vanillic acid to vanillin (Ad/Al)v (0.02-0.87) and syringic acid to syringaldehyde (Ad/Al)s (0.01-0.54) ratios indicate highly degraded lignin materials.

KEYWORDS: Lignin-phenol, monomers, mainland, dissolved organic carbon (DOC), ratio values.

I. INTRODUCTION

The coast of West coast of South Sulawesi is highly vulnerable to anthropogenic runoff, where the region becomes a central settlement with various activities and various spatial functions. Sources of the organic matter from the mainland are transported to the sea through streams and run-off from the mainland [1]. The organic material input loads are present in the form of dissolved organic compounds to particulate organic matter in large aggregates, as well as from the release of humic material and living dead organisms [2,3] and have an impact on the coastal and marine environment, which causes eutrophication (cultural eutrophication) [4,5,6], the possibility of the emergence of dangerous microalgae species [7], damaging coral reef ecosystems and biodiversity [8].

Dissolved organic material is not only a source of energy but also a source of essential organic compounds that can not be synthesized. Much of the organic matter dissolved in seawater consists of complex materials and is highly resistant to bacterial breakdown. Most substances are released by marine life as ectocrines that accelerate or slow growth. Growth is supported by the abundance of humic acid that is ecologically important in coastal waters [9,10]. Various factors and processes affect the distribution of organic materials on various spatial and temporal scales, this condition will create two conditions or systems that often occur in coastal waters [11]. The composition of terrigenous dissolved organic carbon (DOC) is directly to geochemical partitioning within the river estuary [12]. Every year the river flow will bring dissolved organic carbon about 0.25×10^{15} g to the oceans [13].

ISSN: 2277-9655
Impact Factor: 5.164
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A corresponding gradient in freshwater and terrestrially derived inputs of lignin DOC has a characteristic composition that can be attributed to important information sources [14] and it has been documented in biogeochemical processing in riverine/estuarine systems. Numerous studies have examined the humic fraction of terrigenous DOC [15,16].

Lignin is an abundant and stable phenolic macromolecule found in vascular dissolved organic matter in aquatic systems [17,18,19]. The structure of lignin is very diverse depending on the type of plant. Wood or non-timber plants are the main source of lignin that serves as a protector and power giver in plants to withstand mechanical stresses. However, in general lignin is a polymer compound comprising a propane phenyl unit. Most terrigenous organic matter is a humic substance, relatively resistant to microbial degradation [20]. Lignin decomposition takes place very slowly in the environment because of its complex, heterogeneous, water-soluble and aromatic chemical structure [21]. Therefore naturally lignin is difficult to decompose and few microorganisms are able to degrade them [19,20,22]. The stable lignin composition is highly dependent on the source and environmental conditions occurring during the precipitation process [23].

The condition of coastal waters along the west coast of South Sulawesi, which is categorized as productive and there are mangrove ecosystems, seagrass and coral reefs of spermonde, is potentially threatened by the degradation of water quality due to the disposal of organic and inorganic wastes from the land carried by the big rivers in the vicinity [24]. The different content compositions of organic materials in the waste, relative to the marine organic material will help to track the extension of waste in coastal areas. Dissolved lignin analysis has been widely investigated in aquatic systems with different puposes. The objective of this study is to observe the behavior of transportation anthropogenic land using lignin phenols with C/N ratios of terrigenous DOM fraction in to the Spermonde islands.

II. MATERIALS AND METHODS

a. Location and Time of study

The research sites are located in coastal waters by focusing on the estuaries of major rivers, the Tallo coast and Pangkep coast, and some outer islands that represent the gradient of distance and salinity from the mainland (Fig. 1). Water sampling for dissolved lignin measurements, and C/N ratios, and measurements of hydrographic conditions (temperature, salinity, pH and brightness) were carried out during two sea voyages representing the dry season (October 2017) and rainy season (March 2018).

Figure 1 Sampling location

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b. Water Sampling

Water samples were filtered during collection through pre-weighed GF/F 0.7 µm with using the vacuum pump (200 mmHg) and pre-rinsed with at least 3 L of water collected per sample. Samples were kept in the dark and shipped on ice, than immediately transported to the laboratory for dissolved lignin and other analyses.

c. Ctot, Ntot, Ratio C/N and Carbon Isotope Analysis (Elemental and Carbon Isotope Analysis)

1. Organic Carbon Determination (Walkey & Black Method)

The seawater samples were pipetted, then weighed 0.5 grams, then the sample was put into a 100 mL measuring flask, then added 7.5 mL of concentrated H_2SO_4 and 5 mL of $K_2Cr_2O_7$ 1 N solution, then added aquades and shaken until homogeneous. Then left for 15 minutes to cool and added aquadest. Then measured using a UV-Vis spectrophotometer with λ= 561 nm.

2. Total Nitrogen Determination (Kjehdahl Method)

The seawater samples were piped and weighed 0.5 grams, then the sample was put into a Kjehdahl tube and added ±1 gram of selenium mixture and 25 ml of concentrated H_2SO_4 solution and also added 1 gram of Selenium Reagent. Furthermore Kjehdahl tube containing the solution is shaken until all samples wetted with H_2SO_4, then the destruction in the cupboard of acid until saturated. Samples was cooled, then poured into a 100 mL measuring flask and rinsed with aquadest, allowed to cool and then squeezed until the line marks with aquadest and shaken until homogeneous. The shelter was prepared in which 10 mL of H_3BO_3 2% and 4 drops of indicator solution were mixed in Erlenmeyer. After that, pluck the 5 mL of sample solution into the distillation flask. Furthermore, 10 mL of 30% NaOH and 100 mL of aquadest were added. The distillation tip was rinsed with aquadest, then the container together with the contents was titrated with 0.0171 N H_2SO_4 solution.

e. Cupric oxide analysis of lignin phenols

Lignin analysis was carried out in a 250 ml Teflon containers with stainless steel jackets using the cupric oxide (CuO) oxidation method, there are several modifications of [3,14,21]. The seawater sample was piped and weighed 0.5 g, then weighed 0.1 g of $Fe(NH_4)(SO_4)$ 2.6 (H_2O) and 0.5 g of CuO powder added together with 3 mL NaOH 2 N into the mini bomb, and as much as 10-15 mg of glucose to remove the super oxidizing effect. The sample was heated at 175 ^oC for 3 hours. The oxidation product is cooled, then transferred to a centrifuge tube by adding 10 mL of 1N NaOH, then centrifuged at 3000 rpm for 10 minutes. This step is repeated twice. The supernatant was collected from centrifuge treatment 2 times and acidified to pH 1. Furthermore, the supernatant was extracted three times with 10 mL of hexane and included 100 µL of ethyl vanillin 0.5 mg/ml as an internal standard. Excess solvent extraction (hexane) is evaporated by slowly flowing N_2 to obtain dry residues. Further diluted with pyridine standard as much as 300 µL and 150 µL silylating BSTFA solvent containing 1% TMCS (as catalyst) was added. Then the derivatization was carried out by heating at 90 °C for 10 minutes in a closed glass bottle, then allowed to cool. After the sample was cooled, the sample was analyzed by GC-MS Hewlett-Packard 5890 gas chromatograph fitted with an SE30 capillary column (30 m, 0.25 mm internal diameter, SUPELCO).

f. Statistical Analysis

The interaction of season and location factor to lignin phenol composition in waters was analyzed by using variance analysis (ANOVA).

III. RESULTS AND DISCUSSION

a. Conditions of water

Measurements of hydrographic parameters carried out include measurement of pH, temperature, current, salinity and brightness. Coastal waters at the study sites include estuary waters with salinity gradients ranging from 12.7 to 17.8 ‰ in coastal waters of Tallo estuary of Makassar, 12.8-24.1 ‰ in estuary waters of Pangkep River, and 11.2 to 31.9 ‰ in the sea waters. The hydrographic conditions (temperature, pH and brightness) in different seasons showed no significant difference (Table 1). Sea surface temperatures in both seasons are relatively similar, with mean values (± standard deviation) of 30.92 ± 1.97 in the dry season, and 32.06 ± 0.94 in the rainy season. The average pH in the dry and rainy seasons was 7.72 ± 0.22 and 7.60 ± 0.27, respectively. The brightness averaged 46.81 ± 14.09% in the dry season, and 46.54 ± 10.01% in the rainy season, respectively.

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Table 1 The hydrographic conditions

Sampling Time	Location of Coastal Waters	Salinity (‰)	Temperature (°C)	pH	Brightness (%)
Dry season (Oktober 2017)	**Coastal**				
	Tallo Estuary Makassar	12.7-17.8	30.0-32	7.39-7.54	21.7-38.5
	Pangkep Estuary	17.7-24.1	30.5-36.8	7.91-7.98	58.1-67.4
	Sea	14.8-31.9	28-34.9	7..25-8.07	18.4-72.4
Rainy seoson (Maret 2018)	**Coastal**				
	Tallo Estuary Makassar	11.8-14.7	30.8-31.7	6.98-7.88	31.8-33.5
	Pangkep Estuary	12.7-13.8	32.8-34.1	7.25-7.93	44.9-61.1
	Sea	11.2-20.2	30.1-33.4	7.19-7.98	27.6-62.7

b. Bulk parameters: Ctot, Ntot, C / N Ratio and carbon isotope analysis

In this study, the C-N magnitude measurements and the C/N ratio of the dissolved materials (seawater samples) were used to explain the source of terrestrial landfall into the waters. The magnitude and speed of runoff from land is strongly influenced by the season which has implications on Ctot and% Ntot, where the water discharge at the mouth of the river is greater than in the sea. Carbon and Nitrogen content, C/N ratio, carbon isotope signatures and lignin composition are presented in Table 2 and Table 3. Percentages of Ctot and Ntot from minimum to maximum of 0.050-0.142% and 0.016-0.034% respectively in the dry season; 0.417-0.491% and 0.019-0.038% in the rainy season. In Table 2 and Table 3. shows Ctot presentation is more abundant than Ntot from rivers than in marine areas, reflecting the situation of most of the terrestrial organic matter stored before encountering the oceans. Spatial variation has indicated that the organic material input of anthropogenic activity has a more significant influence on the distribution, rather than the natural process. In addition, the temporal factor of season also plays a significant role in accelerating the transport process of terrigenous material, which in the observation season differs significantly at $p<0.5$ The amount of concentration of organic material in soluble form in particular, may also depend on the deposition rate, the nature of the organic source and the amount of flux, the potential for material preservation during transportation, mineralization and degradation [25,26].

Table 2 Organic matter compositions for DOC from the estuary of The River inputs to the ocean (dry season)

Sampling Sites	Ctot (%)	Ntot (%)	[C/N]mol Ratio	$\delta^{13}C$ (‰)	Λ (mg/100mg OC)	S/V	C/V	(Ad/Al)v	(Ad/Al)s
Tallo River Estuary	0.142	0.028	5.07	-7.49	1.63	1.95	0.16	0.49	0.54
Barrang Lompo Island	0.076	0.016	4.75	-6.82	1.48	0.46	0.02	0.37	0.47
Bone Tambung Island	0.107	0.027	3.96	-6.67	1.14	0.13	0.01	0.08	0.29
Langkai Island	0.082	0.023	3.56	-6.43	1.28	0.02	-	0.03	0.01
Lanjukang Island	0.050	0.023	2.17	-2.23	1.14	-	-	0.02	-
Pangkep River Estuary	0.140	0.027	5.18	-5.32	1.57	1.37	0.38	0.36	0.49
Laiya Island	0.131	0.028	4.67	-4.91	1.55	0.35	0.18	0.28	0.33
Sarappo Keke Island	0.132	0.034	3.88	-4.72	1.25	0.11	0.02	0.07	0.31
Kondong Bali Island	0.115	0.031	3.71	-4.64	1.19	0.03	0.01	0.05	0.27
Kapoposang Island	0.058	0.017	3.41	-4.34	1.02	0.02	-	0.02	0.01

The organic C/total N (C/N) ratio from estuary to the oceans is 2.17-5,18% (for dry season) and 12.18-25.57% (for rainy season), respectively. This change in percentage decreases toward the sea, this change indicates that the organic material is derived from terrigenous. The varied C/N ratio of runoff to coastal runoff during the dry season (October) and rain (March) with the range 2-25.5. Table 2 and Table 3 has indicated that the organic

ISSN: 2277-9655
Impact Factor: 5.164
CODEN: IJESS7

material is from the sea and terrigenous (soil organic matter) [27]. The amount of organic material in the form of dissolved from ground overflow, has also helped to track the extension of waste on the west coast of South Sulawesi.

Use of $\delta^{13}C$ values to distinguish sources of organic matter such as marine and significant differences were observed for the freshwater samples. The values of $\delta^{13}C$ from minimum to maximum in each dry season range from -2.23 ‰ to -7.49 ‰ and -9.34 ‰ to -13.49 ‰. The $\delta^{13}C$ values suggested that there is a conservative mix between terrestrial and aquatic sources derived organic matter throughout the entire core. Most vascular plants utilize the photosynthetic pathway of C3, which has a higher value of $\delta^{13}C$ than those utilizing the C4 line. Lignin present in vascular plants can be used as a proxy for organic carbon derived from plants, where lignin results are negatively correlated with $\delta^{13}C$ [28]. This implies that the vascular plants in this system generally come from C3 plants. This applies to soluble, particulate, and sedimentary phases of the freshwater and freshwater systems in which C3 plants predominate [29].

c. Lignin phenols composition

Contributions of three lignin index-phenols (V, S and C) indicating that influxes of the lignin. V-phenols are the most dominant CuO-oxidation products of lignin, followed by S-phenols and C-phenols in the lower parts of the core. In marine samples, the cinnamyl phenol concentrations are not likely to be reported because of significant non-lignin sources [14]. Eight lignin phenols were quantified for all samples. Carbon-normalized yields of lignin (Λ_8), given in units of μg lignin (100 mg OC)$^{-1}$ are often used to estimate the relative contribution of terrestrial organic matter in marine systems. Eight "characteristic" lignin-derived phenolic monomers of the dissolved organic fraction in Tallo and Pangkep river, reflecting both the high abundance of lignin as a component of the terrestrial plant biomass and the preferential accumulation of woody plant fragments (with have a high lignin content) from the estuary.

Λ values in all sampling locations ranged from 1.02 to 1.63 (mg/100mg OC) in the dry season, and 2.15 to 3.14 in the rainy season (Table 2). The product of lignin phenol oxidation in dry season in Tallo waters is higher than in Pangkep waters, as well as lignin phenol composition in the rainy season, but the composition of lignin phenol to the sea is smaller than the horizontal line of the Tallo estuary. The influence of seasonal factor on lignin phenol composition is very significant. There were significant differences between lignin phenol compositions between seasons (drought and rain) and between locations (Makassar and Pangkep). This suggests that the role of seasons in the distribution of lignin phenol dissolves in considerable waters, where the largest composition of lignin phenol in waters occurs in the wet season. The high composition of lignin phenol in rainy season or high precipitation season is also shown in some estuary and coastal aquatic systems in the world although their land characters are different, as in autumn in some estuary areas in the USA, seasons semi in northern Japan [23].

Tabel 3 Organic matter compositions for DOC from the estuary of the River inputs to the ocean (rainy season)

Sampling Sites	OC (%)	N (%)	[C/N] mol Ratio	$\delta^{13}C$ (‰)	Λ (mg/100mg OC)	S/V	C/V	(Ad/Al)v	(Ad/Al)s
Tallo River Estuary	0.486	0.019	25.57	-13.49	3.14	2.29	0.14	0.87	0.37
Barrang Lompo Island	0.433	0.019	22.78	-12.82	2,57	0.75	0.23	0.16	0.28
Bone Tambung Island	0.490	0.026	18.85	-11.67	2.49	0.34	0.07	0.07	0.06
Langkai Island	0.486	0.029	16.75	-11.43	2.25	0.18	0.01	0.01	0.08
Lanjukang Island	0.491	0.031	15.83	-10.61	2.15	0.04	-	0.007	0.03
Pangkep River Estuary	0.442	0.021	21.05	-12.72	2.93	4,32	0.62	0.42	0.28
Laiya Island	0.417	0.022	18..95	-11.95	2.55	1.57	0.21	0.78	0.22
Sarappo Keke Island	0.422	0.024	17.58	-11.69	2.49	0.87	0.06	0.12	0.20
Kondong Bali Island	0.458	0.027	16.96	-11.14	2.42	0.28	0.04	0.23	0.03
Kapoposang Island	0.463	0.038	12.18	-9.34	2.23	0.02	0.03	0.06	0.02

Lignin phenol ratios (S/V) may be changed prior to deposition by a selective degradation of lignin structural units during the transfer of lignin phenol deposits. Syringyl phenols (S) are exclusively found in angiosperms, whereas cinnamyl phenols (C) are present only in non-woody tissues. Therefore, the phenols ratio can be used to

ISSN: 2277-9655
Impact Factor: 5.164
CODEN: IJESS7

distinguish the relative contribution of sources of organic material from plant species or tissues of angiosperms and gimnosperms (S/V). The ratio of C to V phenol was used to differentiate the contribution of woody and nonwoody tissue [14,23,30]. The S/V and C/V ratios are 0.02 to 1.95 and 0.02 to 4.32, respectively, indicating the presence of non-woody angiospermeal plant tissue. The acid/aldehyde ratios of phenol groups have been used to characterize diagenetic alteration in a variety of geochemical samples. The acid: aldehyde ratio values of V-phenols (vanillic acid/vanillin (Ad/Al)v for samples of river estuaries and ocean samples, higher than freshwater samples, indicating that lignin has naturally degraded in marine systems. (Ad/Al)v is a better indicator of lignin diagenesis than (Ad/Al)s because there are usually higher levels of vanillyl oxidation products. The values of (Ad/Al)v in all sampling locations during the dry season ranges from 0.002 to 0.49, while in the rainy season, range from 0.007 to 0.87. The value of syringic acid to syringaldehyde (Ad/Al)s in dry and rainy seasons ranged from 0.01 to 0.54 and 0.02 to 0.37 respectively.

IV. CONCLUSION

This study shows that the soluble lignin phenol composition in coastal waters of South Sulawesi is relatively small. The difference of lignin phenol composition between Makassar and Pangkep waters to some outermost islands which represent the distance gradient of mainland is more influenced by season factor, where the composition of soluble phenol lignin is greater in the rainy season. Makassar waters contain average lignin phenol which is bigger than Pangkep waters this explains that the significance of spatial character of each waters. The lignin phenol monomer starts from the Tallo estuary and Pangkep up to some outer islands, indicating the composition of lignin phenol is getting smaller. However, although lignin phenol composition is larger in Makassar waters, lignin phenol monomers in outer islands are undetectable. This condition explains that lignin has been degraded to some outer islands. Rapid decomposition of organic matter in the middle river is also facilitated by the improvement of fresh phytoplankton and terrestrial organic matter. This will make both old and young organic matter is degraded. The densely populated areas along the banks of the Tallo and Pangkep River have led to an increase in refractory of some peat material, as anthropogenic OC.

V. ACKNOWLEDGEMENTS

The research was funded by a Doctoral Dissertation Grant from the Ministry of Research and Technology - Indonesia in 2018. The authors would like to thank or all those have assisted this research.

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ISSN: 2277-9655
Impact Factor: 5.164
CODEN: IJESS7

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CITE AN ARTICLE

Rustiah, W., Noor, A., M., & Lukman, M. (2018). ASSESSMENT OF TRANSPORT MATERIAL MAINLAND BASED ON THE COMPOSITION OF LIGNIN PHENOLS IN COASTAL WATER OF THE SPERMONDE ARCHIPELAGO, INDONESIA. *INTERNATIONAL JOURNAL OF ENGINEERING SCIENCES & RESEARCH TECHNOLOGY*,*7*(5), 446-453.