ISSN 0853 – 2788
Akreditasi No : 345/Akred-LIPI/P2MBI/07/2011

POTENTIAL OF COGON GRASS AS AN OIL SORBENT

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Diterima : 30 April 2012; Disetujui : 25 Mei 2012

ABSTRACT

Experiments on the potential of Cogon grass (Imperata cylindrica), a weed harmful to other plants, for use as a low-cost and biodegradable oil sorbent were carried out under various spill conditions. Flowers of Cogon grass adsorbed much larger amount of high-viscosity lubricating oil (57.9 g-oil/g-sorbent) than that adsorbed by Peat Sorb (7.7 g-oil/g-sorbent), a commercial oil-sorbent based on peat. However, the flowers adsorbed only 27.9 g of low-viscosity crude oil/g-sorbent. In an oil–water system, the amount of oil adsorbed was influenced by the level with which the two were mixed: vigorous stirring reduced the sorption capacity by 36%. The high sorptive capacity of the flowers can be attributed to their hydrophobic nature and good oil-wettability. The flowers showed good buoyancy even after 24 hours of shaking under conditions that simulated water ripple (gentle wavy motion) in sea, which suggests their potential in combating oil spills both on land and in water.

***Keywords**: Sorbent; Oil spill; Oil sorption; Cogon grass; Imperata cylindrica*

ABSTRAK

Telah dilakukan pengujian terhadap potensi bunga rumput Alang-alang (*Imperata cilindrica*) sebagai sorben penyerap-minyak pada berbagai kondisi tumpahan minyak di lahan kering dan di air. Pada permukaan yang kering, bunga rumput Alang-alang dapat menyerap jauh lebih banyak minyak pelumas (57,9 g-minyak/g-sorben) dibanding Peat Sorb (7,7 g-minyak/g-sorben), contoh sorben komersial berbasis gambut. Sedangkan terhadap minyak mentah dengan viskositas rendah, bunga rumput hanya menyerap sebanyak 27,7 g-minyak/g-sorben. Pada penanganan tumpahan minyak di air, jumlah minyak yang dapat diserap dipengaruhi oleh tingkat pengadukan minyak dan air. Pengadukan yang kuat dapat menurunkan kapasitas penyerapan minyak hingga 36% dibanding tanpa pengadukan. Kapasitas penyerapan bunga Alang-alang yang tinggi ini dipengaruhi oleh sifat hidrofobisitasnya yang baik. Bunga Alang-alang juga menunjukkan sifat mengambang yang baik pada permukaan air yang diguncang menyerupai riak air di laut. Hasil diatas menunjukkan bahwa bunga alang-alang berpotensi baik sebagai bahan sorben penyerap-minyak untuk penanganan tumpahan minyak di lahan kering dan di air.

***Kata Kunci**: Sorben, Tumpahan minyak, Penyerapan minyak, Rumput Alang-alang, *Imperata cylindrica*.*

INTRODUCTION

Oil spills are one of the major sources of environmental pollution in land and marine environments. Improving the techniques for controlling and removing oil spills is an active area of research. One of the approaches to control is to develop oil sorbents that can remove oil from a spill site completely. Based on the nature of raw materials, oil sorbents can be grouped into three major classes, namely inorganic minerals, synthetic organics, and natural organics. A comprehensive review of this subject can be found in Adebajo et al.(1). Commercial oil sorbents commonly used by many petroleum companies are synthetic sorbents made of polypropylene and polyurethane. They have good hydrophobic and oleophilic properties, but their non-biodegradability is a major disadvantage, particularly because they need to be disposed of properly after use. This limitation has led the search for alternative methods using biodegradable material such as lignocellulosic fibers of agricultural residues or weeds. In many cases, the material is locally and cheaply available, the main costs being mainly those of collections and preparation. It is much easier to compost lignocellulosic fibers than synthetic polymers after use[2], which reduces the hazard and cost associated with incineration or other means of disposal. Moreover, cellulosic products exist in fibrous form and can be easily made into mats, pads, or nonwoven sheets[3]. A number of natural sorbents have been studied for use in cleaning up oil spills. Some of them have good oil sorption capacity, but they also adsorb water at the same time, which is a disadvantage when they are used in aqueous environment.

Several studies to find sorbent materials from natural organics with high oil sorption capacity, high hydrophobicity, and good buoyancy are described below. Basically they can be grouped into (1) explorations of new plant material and (2) chemical treatment of known plant material. Choi and Cloud[4] found that milkweed floss (*Asclepias*) fiber adsorbed significantly larger amount of crude oil (approximately 40 g oil/g fiber) than such artificial fibers as nylon, polyester, acetate, viscose rayon, or polypropylene (typically below 25 g/g)[5]. Nonliving biomass of *Salvinia* sp. was identified to adsorb only 4.8 g crude oil/g biomass[6], and Suni et al.[7] reported that cotton grass (*Eriophorum vaginatum*) removed up to 20 times its own weight of diesel oil. Recently, Annunciado et al.[8] found silk floss (*Chorisia speciosa*) fibers to have a very high oil sorption capacity (approximately 85 g/g). Meanwhile, other research groups have been trying to make some plant material more hydrophobic by chemical treatment: wood bark saturated with transition metal ions[9-11], acetylation of cotton fiber[12,13], rice straw[14], and sugarcane bagasse[15]. Although these chemical treatments significantly improved sorption capacity, the result was a more expensive sorbent product. Other important factors that need to be considered are local availability and abundance of the sorbent materials in the area of spills, particularly in remote areas where options to transport bulky material to the site are limited. Therefore, identifying such material for use as a clean-up tool for oil spills is of significant commercial interest.

Cogon grass (*Imperata cylindrica*) was ranked as one of the ten worst weeds of the world particularly because it can spread, colonize, and subsequently displace other desirable vegetation[16]. Cogon grass regenerates very rapidly from its underground rhizomes and responds with flowering to such control measures or forms of stress as burning, overgrazing, drought, and repeated slashing[17]. It grows in various ecosystems from the dry to the moist natural areas, and has been reported as a weed in 73 countries, mostly in Africa, Australia, southern Asia, and the Pacific[16]. This paper discusses the potential use of Cogon grass as oil sorbent in cleaning up oil spills on land and in water.

MATERIAL AND METHODS

Sorbent material

The grass was obtained from a plot of marginal land in Serpong (Banten, Indonesia). A commercial natural oil sorbent (Peat Sorb), a processed peat, provided by a sales agent in Jakarta, was used for comparison. The grass was air-dried, its flowers (spikelets) were separated from the stem, and the stem was cut into pieces 2–4 mm long. Peat Sorb was ground in a mill (Quacker City Mill model 4-G, Philadelphia, USA) and sieved in a laboratory test sieve (Retsch, Germany) to obtain particles of 500–600 μm. All such material was stored in a dessicator; it was removed from the desiccator and aired overnight at room temperature before use. Moisture content of the grass flowers after such pretreatment was typically 6%–8%.

To quantify buoyancy, 25 mg of grass flowers was placed in a 1000 mL glass beaker containing 500 mL water or a mixture of 450 mL water and 50 mL oil and the contents stirred for 60 min at 72 rpm. At the end of the test, the part that remained afloat was removed, dried, and weighed to calculate the buoyancy percentage.

Specific gravity of the grass flowers was measured using a picnometer with hexane (SG =

0.66) as the test fluid. This non-polar solvent was used because of its low specific gravity and because it apparently did not react with the flowers.

The BET (Brunauer, Emmett, Teller) value for the surface area for nitrogen sorption was determined using a Nova 1000 Quantachrome. This method is often used as an indicator of the degree of microporosity of sorbents. The measurements were taken after the grass flowers had been immersed in liquid nitrogen and ground to powder in a mortar, as BET suface area cannot be detemined directly for large particles.

Wettability of the grass flower by both oil and water was estimated by the speed and extent to which each liquid rises in a packed column of the material, known as the Washburn test[18]. Hexane represented a non-polar solution and water, a polar solution. The method described by Ribeiro et al.[6] was followed with minor modifications. The grass flowers were packed manually in a glass tube (50 cm long with an inner diameter of 8 mm) closed at one end with a 1-cm-thick plug of cotton. Approximately 1.5 g of the grass flower was needed to fill 44 cm of the tube. This column was then dipped in a 1000-mL beaker containing 300 ml of hexane or water, and the level of liquid rise inside the tube was recorded as a function of time. The zero time was started when the level of the liquid in the tube reaching the level of the liquid inside the beaker.

To measure the area of a cross section of a broken fiber of the grass flower, a scanning electron microscope (Philips 515) was used. The specimen was prepared by immersing the material into liquid nitrogen and grinding it to powder in a mortar. Gold-coated samples were then mounted on a universal base and placed in a vacuum chamber at a high pressure (1.333 10^{-2} Pa or 10^{-4} Torr).

Test oils

The following oils were used for the sorption tests: light crude oil (Cemara crude oil), automotive diesel oil (Solar), lubricating oil (Mesran Super 20W-50) obtained from Pertamina (a major Indonesian petroleum company), and heavy crude oil (Petani crude oil) obtained from PT CPI (Chevron Pacific Indonesia). Kinematic viscosity was determined at 40 °C by a viscometer (Koehler Instruments, model K-23429, Germany) and specific gravity was measured using a picnometer (Duran, 25 mL and 10 mL). In order to know the tendency of oil to evaporate, oil samples were placed in a glass dish 9 cm in diameter and 1.5 cm deep, and the loss in weight after 24 hours of storage at room temperature and pressure was recorded.

Sorbents are generally not effective in cleaning up highly viscous or heavy oils. These oils do not adhere to the sorbent readily owing to their poor wettability; on contact with water, highly viscous oils tend to sink partly and heavy oils tend to sink completely. The oil should not be highly volatile either lest it should evaporate quickly. The physical characteristics of the oils used in the experiment are shown in **Table 1**.

Table 1. Oil types and their properties

Oil types	Specific gravity	Kinematic viscosity (cSt)[a]	Weight loss (%)[b]
Petani crude oil	0.865	35.43	2
Cemara crude oil	0.824	1.90	20
Diesel oil	0.840	4.15	2
Lubricating oil	0.884	175.60	0

[a] *viscosity at 40 °C*
[b] *after 24 hours at room temperature*

All the oils had a specific gravity lower than unity, and therefore they would not sink in water. At 30 °C, their viscosity was low enough so for them to adhere to the sorbents easily, with the exception of Petani crude oil, which had to be warmed to 35 °C for it to be fluid enough to be adsorbed. Data on oil volatility show that during 20 minutes of the sorption experiment, the weight loss due to evaporation of oil was less than 0.3% of the original weight. The above properties indicate that these oils are suitable to be removed using sorbent as a clean-up tool.

Sorption experiments

To assess the capacity of the biomass to adsorb oil in absence of water (dry system), a procedure specifically set for loose particulate sorbents was used[19]. The sorbent (0.25 g for grass and 1 g for Peat Sorb) was placed inside a wire basket (pore size 150 μm), made of steel wire, 6.2 cm in height and 4.5 cm in diameter. The basket was then dipped in a 1-liter beaker containing 500 ml of oil. Because it is bulky, smaller amount of grass flower was used in this experiment. The beaker was in turn placed in an agitator (flocculation tester, SBS Instruments) and the liquid stirred at 100 rpm for 20 minutes, considered long enough for the sorbent to be saturated with oil. The basket containing the sorbent was then raised, the oil allowed to drain by gravitation for 1 minute, and the basket weighed. An empty basket, subjected to the same process to account for oil deposited onto metal surfaces, functioned as a blank. The amount of oil adsorbed was measured as the weight gained after sorption, expressed in grams per gram of dry sorbent. All sorption tests were run at 30 °C ± 0.5 °C, except those for Petani crude oil, which were run at 35°C ± 0.5°C because the oil is highly viscous. These temperatures were chosen to represent ambient temperature in the tropics.

In oil–water systems, only two types of oils (lubricating oil and Cemara crude oil) were further tested to represent high- and low-viscosity oils respectively. The experiments were carried out at three levels of mixing (static, gentle, and vigorous) in a 1000 mL glass beaker containing 450 mL tap water and 50 mL oil. For static condition and gentle mixing, the grass flowers were poured onto the surface of the oil and, after saturation, collected in the metal basket described above. For static condition, the contents were not mixed at all; for gentle mixing, intended to simulate waves in the marine environment, the beaker was shaken in an orbital shaker (Cole Parmer 51300-05) at 72 rpm. Vigorous mixing, to simulate the environment in which the oil is completely dispersed in water, was carried out in a Dynamic Heidolph MR 2002 mixer and a stirring bar operating at 1000 rpm; the sorbent remained in the basket, the same as in dry oil system. The apparatus was observed every 20 minutes over an hour to see the effect of contact time on the amount of oil adsorbed. The sorbent saturated with oil was then raised to allow the excess oil to drain by gravitation for 4 minutes, and weighed. Longer time was provided for draining in the oil–water system than the dry system to make sure that the sorbent was free of any drops of liquid adhering to it. Water was extracted from saturated sorbent with fluids as described in ASTM[20], using a Dean-Stark glass still, with a mixture of xylene and toluene (80:20, v/v) as a solvent. The results of all measurements were presented as average of three replicates. The bars denote standard deviation of the replicates.

RESULTS AND DISCUSSION

Characteristics of the Cogon grass flower

The grass was identified at Herbarium Bogoriense, Indonesian Institute of Sciences, as ***Imperata cylindrica*** (L.) Beauv, known as Alang-alang or Ilalang (Indonesian) or Cogon grass (English).

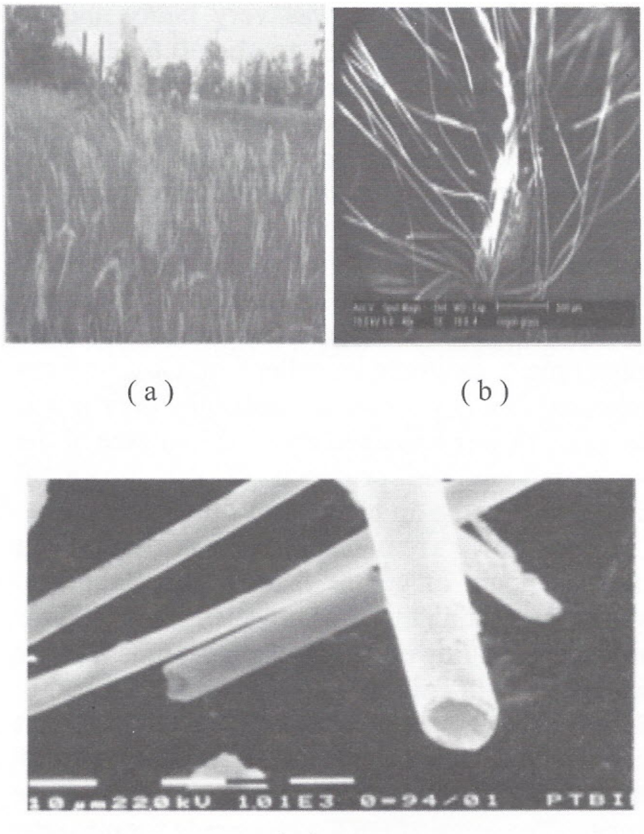

Figure 1. Cogon grass and its hairy flower: (a) Cogon grass growing on marginal land, (b) grass flower at ×40 magnification, (c) cross-section of the rod-like grass flower fiber at ×1000 magnification showing the fiber's hollow structure.

Figure 1 shows the morphology of the grass and a magnified image of flower parts. Figure 1a shows Cogon grass growing on a piece of marginal land. The plant is a slender, flat, linear-lanceolate stemless grass, less than a meter tall, arising from a thick underground mat of rhizomes. A complete morphological description of this grass can be found in Aguilar[21]. **Figure 1b** shows the characteristics of a single hairy flower (spikelet) about 8–10 mm long. Its snowy white color suggested a waxy coating. The importance of the waxy surface to the hydrophobic and oleophilic properties of cotton and milkweed was reported by Choi and Cloud(4); it is believed to confer similar properties on Cogon grass. **Figure 1c** shows the hollow structure (lumen) of the hairy parts of the Cogon grass flower. The same kind of rod-like fiber found in kapok, milkweed, and cotton has been demonstrated to contribute to the high oil-sorption capacity owing to increased surface area and enhanced capillary action(5).

The grass flower was very bulky and its density was estimated to be 0.66 g/cm^3, approximately the same as that of hexane in which the grass flower dispersed evenly in the liquid phase. After saturation with oil, the volume of the grass flower is usually smaller. Therefore, in its application as oil sorbent, this material can be compacted to some extent, but not so much that little space is left for the oil. After saturated with oil, the sorbent needs to remain buoyant in water for some time to facilitate its collection. Observations indicated that in the beaker containing only water, the grass flower remained above the surface of the water but in the presence of oil, it would concentrate in the oil phase above the water phase. The grass flowers remained in these positions even after 24 hours of gentle shaking that simulated waves, suggesting that the buoyancy was 100% for both the grass flower in water and the grass flower saturated with oil in the oil–water mixture. Buoyancy over such a long period is an important feature of the Cogon grass flower, making it easier to collect the grass and remove it from water bodies. The value of BET surface area for nitrogen sorption of the grass flower was estimated to be 1.84 m^2/g. This value was obtained for the grass flower in a powder form, as BET suface area for large particle cannot be detemined directly.

Wettability of the grass flower in oil or in water is an important parameter when the sorbent is to be used in aquatic environment. A good oil-sorbent should adsorb a great deal of oil but only a small amount of water. Wettability can be measured in terms of the level of fluid penetration in a column filled with powder or fiber. According to Perry and Green(18), the capillary action in such a system is controlled by the viscosity of the fluid, capillary radius, surface tension between the liquid and air, contact angle between the liquid and solid surfaces, and time. In addition, Choi and Moreau(5) demonstrated that capillary action took place not only in the internal lumen of a hollow fiber but also within the voids (capillary bridges) between fibers. In our experiment, both mechanisms can be expected to operate because of the presence of the hollow structure of the grass flower fiber shown in ***Figure 1c***. Hexane, as a non-polar organic solvent, was used to represent various oils tested, while water represented the polar fluid.

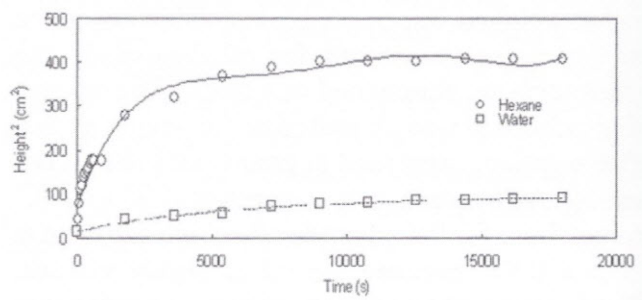

Figure 2. Hexane and water capillary rise in a tube packed with Cogon grass flower.

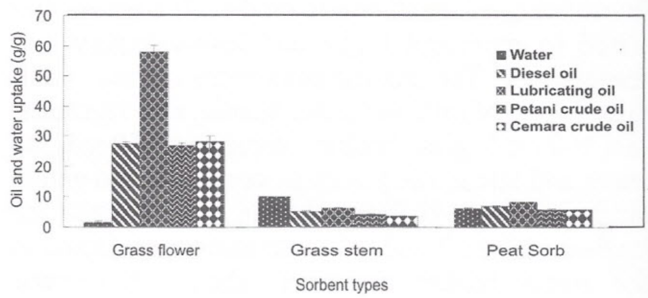

Figure 3. Sorption of water and oil by Cogon grass flower, flower stem, and Peat Sorb in water-only and oil only systems.

A large difference in the level of penetration of the fluids in the column between hexane (h^2 = 400 cm^2) and water (h^2 = 90 cm^2) showed that the grass flower is wetted far more easily by hexane than by water **(Figure 2)**, indicating that the grass flower has good wettability in oil and is hydrophobic and supporting the results shown in **Figure 3**, namely that the grass flower adsorbed as much as 57.9 g oil/g but only a small quantity of water (1.5 g/g). This oil sorption capacity is much higher than that of milkweed floss (40 g/g) reported by Choi and Cloud(4).

Effect of sorbent materials

Results of the experiment with the dry oil system, i.e. sorption characteristics of different sorbents in an oil bath without the presence of water, are given in **Figure 3**. The experiment was meant to simulate the cleaning up of an oil spill on dry land. The flower parts of the grass adsorbed more lubricating oil (57.9 g/g) than that adsorbed by the stem (5.9 g/g) or by Peat Sorb (7.7 g/g), a commercial natural oil sorbent based on peat. Other oils (diesel and crude oils) were adsorbed in smaller amounts. In another part of this paper, it will be shown that sorption preferences of some oils for different sorbents are somehow related to viscosity. The greater adsorptive capacity of the grass flower than the stem or Peat Sorb suggested that the surface area of the flowers was much larger than that of the other two. Also, the grass flower was more hydrophobic, retaining the least amount of water (1.5 g/g), compared to the stem (9.2 g/g) or Peat Sorb (5.9 g/g). Hydrophobicity is an important quality required of an effective sorbent for its application in aquatic environment. Ribeiro et al.[6] and Annunciado et al.[8] used the same commercial product, Peat Sorb, for comparison. They found a rather wide range of oil sorption capacity, from 2.7 to 9.8 g/g, and our result falls within this range. These differences occured possibly due to variations in particle sizes of the pretreated Peat Sorb and the types of oil tested.

Effect of mixing in oil–water systems

Experimental results in the dry oil system and in the oil-in-water system for high-viscosity oil (lubricating oil) and low-viscosity oil (Cemara crude oil) are summarized in **Figures 4 and 5** respectively.

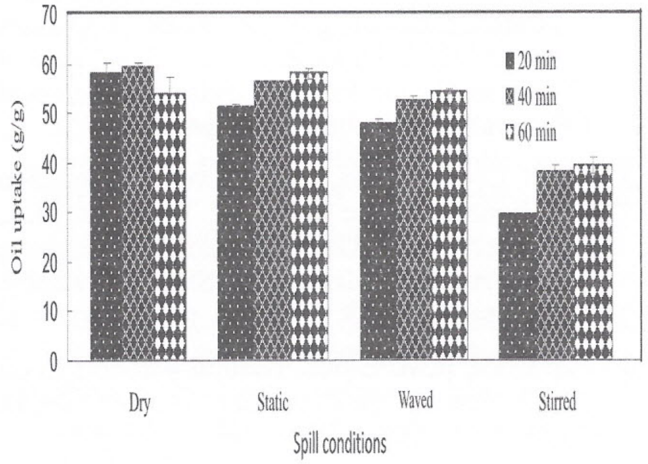

Figure 4. Sorption of high viscosity lubricating oil by Cogon grass flower under various spill conditions. Dry = oil only, stirred at 100 rpm; Static = oil in water, no mixing; Gentle Mixing = oil in water, shaken at 72 rpm; Vigorous Mixing = oil in water, stirred at 1000 rpm. 20, 40, 60 mins denote sorption period.

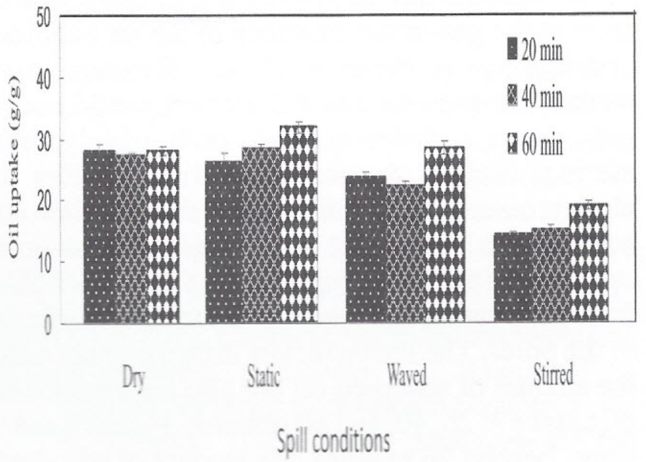

Figure 5. Sorption of low viscosity crude oil by Cogon grass flower under various spill conditions. Dry = oil only, stirred at 100 rpm; Static = oil in water, no mixing; Gentle Mixing = oil in water, shaken at 72 rpm; Vigorous Mixing = oil in water, stirred at 1000 rpm. 20, 40, 60 mins denote sorption period.

The highest amount of lubricating oil was adsorbed in the dry oil system; in the presence of water, the amount of oil adsorbed decreased slightly with static condition and gentle stirring, but significantly (up to 36%) with vigorous stirring. Similar trends were observed for Cemara crude oil. The dry-oil system adsorbed more oil because in this case, most of the surface area of the grass flower was available for the oil whereas in the oil–water system, there was competition between water and oil for the same surface areas, and parts of them would be occupied by water. These differences, however, were not significant, because the grass flower, being highly hydrophobic, adsorbed very small amount of water (**Figure 3**).

The different mixing regimes in the oil–water system affected the amount of oil adsorbed. The static system was a two-phase system where no mixing was involved; gentle mixing involved shaking at 72 rpm; and vigorous mixing involved stirring at 1000 rpm. As indicated above, only vigorous stirring significantly reduced the amount of oil adsorbed (by 36%). This can be explained as follows. The mixing enhanced mass transfer of air into the liquid phase and increased dispersion of air bubbles. However, more vigorous stirring lead to more air bubbles, resulting in increased resistance on part of the oil to attach to the surface of the sorbent. In the static system or gently stirred system, there were no air bubbles, and oil was directly in contact with the sorbent all the time.

In the vigorous stirred system, a film of oil would form at the gas–water interface of the air bubbles, constituting a discrete phase. Consequently, contact between oil and the sorbent would occur only when turbulence (the result of vigorous stirring) brought the sorbent and air bubbles in close proximity. In addition, the diameter of the air bubbles was likely to be much larger than the pore openings of the sorbent, preventing the air bubbles from penetrating and reaching the internal surfaces of the pores. The above factors influenced not only the amount of adsorbed oil but also the kinetics of oil uptake at 20, 40, and 60 minutes. Figures 4 and 5 show, beyond 20 minutes, the amount of adsorbed oil increased only slightly. Studies by Annunciado et al.[8] also confirm that further observation up to 24 hours did not result in any significant increase in oil uptake because the sorbent had almost reached its saturation equilibrium by 20 minutes.

Effect of oil viscocity

Whereas adsorption of oil onto sorbent surfaces is dominated by hydrophobic interaction and van der Waals force, absorption takes place within the porous matrices by diffusion[4]. Besides, as previously mentioned, capillary movements would also take place whenever the sorbent material is in the form of fibers. All of the above sorption mechanisms (wettability, diffusion in porous matrices, and capillary action) are known to be affected by oil viscosity. In this study, within the range of viscosity tested, the amount of oil adsorbed by the grass flower suggests a direct relation to oil viscosity at the temperature of contact. Cemara crude oil, the least viscous (1.9 cSt), was adsorbed the least (27.9 g/g) whereas lubricating oil, the most viscous (175.6 cSt), was adsorbed the most (57.9 g/g).

CONCLUSIONS

It is concluded that the characteristics of the sorbent material, nature of oils, and spill conditions influence the effectiveness of oil sorbent. Cogon grass (***Imperata cylindrica***) proved to be an excellent oil-sorbent under different spill conditions. In addition to low cost, the grass is more environment-friendly because it is relatively biodegradable whereas synthetic polymers, the commonly used alternative, are not. Grass flowers showed high sorption capacity for various crude oils and oil products because the flowers were highly hydrophobic and easily wetted by oil. The grass material also showed good buoyancy even after 24 hours of shaking that simulated sea waves, suggesting the material's potential in combating oil spills not only on land but also in water.

ACKNOWLEDGEMENTS

The experiment was carried out at the Research Center for Chemistry, LIPI, Puspiptek, Serpong. The authors would like to thank BPH MIGAS, PERTAMINA Jawa Barat and PT CPI Jakarta for providing oil samples; also, Dian and Rocky for laboratory assistance.

REFRENCES

1. M.O. Adebajo, R.L. Frost, J.T. Kloprogge, O. Carmody, S. Kokot. Porous materials for oil spill cleanup: a review of synthesis and absorbing properties. *J. Porous Mat.* 10: 159-170 (2003).
2. K.S. Ro, G.A. Breitenbeck, A. Ghalambor. Composting Technology for Practical and Safe Remediation of Oil Spill Residuals, Louisiana Oil Spill Coordinator's Office/Office of the Governor, Louisiana Applied Oil Spill Research and Development Program, Baton Rouge, Louisiana, Technical Report Series 97-009, 1998, 44 p.
3. B.G. Lee, J.S. Han, R.M. Rowell. Oil sorption by lignocellulosic fibers, in: T. Sellers, N.A. Reichert (Eds.), *Kenaf Properties, Processing and Products*, Mississippi State University, 1999, pp. 423-433.
4. H. Choi, R.M. Cloud. Natural sorbents in oil spill cleanup. *Environ. Sci. Technol.* 26(4): 772-776 (1992).
5. H. Choi, J.P. Moreau. Oil sorption behavior of various sorbents studied by sorption capacity measurement and environmental scanning electron microscopy. *Microsc. Res. Techniq.* 25: 447-455 (1993).

6. T.H. Ribeiro, R.W. Smith, J. Rubio. Sorption of oils by the nonliving biomass of a Salvinia sp. *Environ. Sci. Technol.* 34: 5201-5205 (2000).

7. S. Suni, A.L. Kosunen, M. Hautala, A. Pasila, M. Romantschuk. Use of a by-product of peat excavation, cotton grass fibre, as a sorbent for oil-spills. *Mar. Pollut. Bull.* 49: 916-921 (2004).

8. T.R. Annunciado, T.H.D. Sydenstricker, S.C. Amico. Experimental investigation of various vegetable fibers as sorbent materials for oil spills. *Mar. Pollut. Bull.* 50(11): 1340-1346 (2005).

9. M. Haussard, I. Gaballah, P.D. Donato, O. Barres. Removal of hydrocarbons from wastewater using treated bark. *J. Air Waste Manage.* 51: 1351-1358 (2001).

10. M. Haussard, I. Gaballah, P.D. Donato, O. Barres. Use of treated bark for the removal of lipids from water. *J. Air Waste Manage.* 52: 76-83 (2002).

11. M. Haussard, I. Gaballah, N. Kanari, P.D. Donato, O. Barres, F. Villieras. Separation of hydrocarbons and lipid from water using treated bark. *Water Res.* 37: 362-374 (2003).

12. G. Deschamps, H. Caruel, M.E. Borredon, C. Bonnin, C. Vignoles. Oil removal from water by selective sorption on hydrophobic cotton fibers. 1. Study of sorption properties and comparison with other cotton fiber-based sorbents. *Environ. Sci. Technol.* 37: 1013-1015 (2003).

13. M.O. Adebajo, R.L. Frost. Acetylation of raw cotton for oil spill cleanup application: an FTIR and 13C MAS NMR spectroscopic investigation. *Spectrochim. Acta A* 60(10): 2315-2321 (2004).

14. X.F. Sun, R.C. Sun, J.X. Sun. Acetylation of rice straw with or without catalysts and its characterization as a natural sorbent in oil spill cleanup. *J. Agr. Food Chem.* 50(22): 6428-6433 (2002).

15. X.F. Sun, R.C. Sun, J.X. Sun. A convenient acetylation of sugarcane bagasse using NBS as a catalyst for the preparation of oil sorption-active materials. *J. Mater. Sci.* 38(19): 3915-3923 (2003).

16. A.N. Van Loan, J.R. Meeker, M.C. Minno. Cogon grass, in: R. Van Driesche, S. Lyon, B. Blossey, M. Hoddle, R. Reardon (Eds.), *Biological Control of Invasive Plants in the Eastern United States*, USDA Forest Service Publication FHTET-2002-04, Morgantown, West Virginia, 2002, pp. 353-364.

17. D. Chikoye. Characteristics and management of *Imperata cylindrica* (L.) Raeuschel in smallholder farms in developing countries, in: R. Labrada (Ed.), *Weed Management for Developing Countries (Addendum 1)*, FAO Plant Production and Protection Papers, Rome, 2003, 290 p.

18. R.H. Perry, D.W. Green. *Perry's Chemical Engineers' Handbook*, seventh ed., McGraw-Hill, New York, 1997.

19. ASTM F726-82. *Standard Method of Testing Sorbent Performance of Adsorbents*, American Society of Testing and Materials, Philadelphia, PA, 1994.

20. ASTM D95-83. *Standard Test Method for Water in Petroleum Products and Bituminous Materials by Distillation*, American Society of Testing and Materials, Philadelphia, PA, 1990.

21. N.O. Aguilar. *Imperata cylindrica* (L.) Raeuschel in: L. 'tMannetje, R.M. Jones (Eds.), *Plant Resources of South-East Asia No 4: Forages*, PROSEA Foundation, Bogor, Indonesia, 1992, pp 140-142.Jatamason in Polyherbal Formulation, International Journal of ChemTech Research CODEN (USA), 1 (4), 1079-1086.

19. M.D. Phale, P.D. Hamrapurkar, M.A. Chachad, 2010, Precise and Sensitive HPTLC Method for Quantitative Estimation of Wedelolactone in Eclipta Alba Hassk, Pharmacophore, 1 (2), 103-111.

20. N.S. Wani, T.A. Desmukh, V.R. Patil, 2010, A Rapid Densitometric Method for Quantification of Wedelolactone in Herbal Formulation using HPTLC, J. Chem. Metrol. 4(1), 28-33.

21. K. Savita, K. Prakashchandra, 2011, Optimization of Extraction Conditions and

Development of Sensitive HPTLC Method for Estimation of Wedelolactone in different extracts of Eclipta alba, International Journal of Pharmaceutical Sciences and Drug Research, 3(1): 56-61.

22. M.N. Prajapati, T.A. Deshmukh, V.R. Patil, B.K. Shrikhande, 2011, Development and Validation of HPTLC Method for Simultaneous Determination of Wedelolactone and Phyllanthin in Hepasuport Tablet, Der Pharmacia Sinica, 2(4):140-147.

23. H. Yuan, Yunli, Zhao, Xiaoying, Wang, Qian Tang, Xiaoxia Gao, Zhiguo Yu, 2007, Simultaneous determination of *wedelolactone* and isodemetil-*wedelolactone* in herba eclipta by reverse-phase high performance liquid chromatography, Se Pu, 25(3), 371-373.

24. K. Danzer 2010, Analytical Chemistry, Theoretical and Metrological Fundamentals, Springer-Verlag, Berlin Heidelberg.

25. AOAC, 2000, Official Methods of Analysis of AOAC International, 17 th ed, AOAC International, Gaithersburg, Maryland, USA.

26. ICH-Q2B, 1996, Validation of Analytical Procedures: Methodology, Guidance for Industry, International Conference of Harmonization, Geneva.

27. T. Farrant, 1997, Practical Statistic for the Analytical Scientist, The Royal Society of Chemistry, LGC Teddington, Cambridge.