Comparison of raw rice husk and raw rice straw (*Oryza sativa* L.) for diesel oil removal

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Abstract

Oil spillages result in detrimental environmental hazards, posing mutagenic and carcinogenic effects. Targeting oil removal and repurposing of agricultural wastes, this study aims to assess and compare the percent sorption of raw rice husk (RRH) and raw rice straw (RRS) for pure diesel oil. A tetrahedral framework in the packing of the sorbents was incorporated to control their bulk density. Results showed that RRH, RRS, and sorbent pad have mean percent sorption of 2.97%, 4.71%, and 7.25%, respectively. The significantly higher mean percent sorption of RRS than RRH can be linked to their porosity, surface roughness, and presence of functional groups and lignocellulosic components. Thus, RRS can better sorb diesel oil than RRH. Although it is not comparable with the commercially-used sorbent pad, the use of RRS is still recommended as an alternative sorbent for oil spill clean-up.

Introduction. - Oil spills may contribute environmental damage to lands and seas that could necessitate years or decades of recovery due to the release of toxic hydrocarbon compounds known as polycyclic aromatic hydrocarbons [1,2]. Among reported spill incidents, diesel constitutes 20% of the encountered oil type that originates from major sources such as power plants and power barges [3]. Consequently, diesel oil, with a density of 0.81-0.84 g/cm^3, has been recognized as one of the relevant oils that require oil spill response in the Philippines [3]. Thus, diesel, exhibiting better diffusion and higher penetration in sorbents, has been utilized for evaluating the adsorptive characteristics of sorbents [4,5]. Specifically, the diesel used in this study contains a mixture of paraffins, cycloparaffins, aromatic and olefinic hydrocarbons, and has a density of 0.84 g/cm^3 [6].

Various technologies for the removal of organic pollutants have been explored by previous studies, such as oxidation, reverse osmosis, ion exchange, electrodialysis, electrolysis, and adsorption [7]. Among these, sorption, constituting both absorption and adsorption processes, was widely used due to its wide availability, low cost, and ease of operation [7,8]. It refers to the mechanism dependent on sorbent porosity by which a sorbate is incorporated into the porous structures of a material [9]. Other processes involve splitting the compounds into their basic elements and utilizing pressure or electric energy for oil removal.

With the increased amount of non-edible biomass, organic sorbents have been targeted in oil spill treatments, owing to their low cost, high availability, good biodegradability, and other eco-friendly properties [9,10,11,12]. Sorption capabilities of rice husk and rice straw have been investigated in various sorbates such as dyes, light crude oil, and crude, spent, and engine oil [13,14,15]. Modifications of sorbents have been employed including alkali treatment, thermal modifications, acetylation, and carbonization [1,5,17,18,19,20]. However, these pre-treatment processes were contradicted as time-consuming and costly [18]. Therefore, researchers utilized the raw states of the sorbents. On the other hand, the sorbent pads obtained for this study meet the standards set by the industry for managing spills.

The properties of the sorbents promote their sorption capabilities. The void spaces in the matrices contribute to their porosity, while the composition such as cellulose, hemicellulose, and lignin account for their hydrophobic-oleophilic properties [10,19]. These properties allow the attachment and entrapment of oil within their structures [21]. However, grinding causes damage to the pores, which hinders oil attachment on its surfaces [15]. Thus, the use of raw unground materials appears to be more desirable in emergency oil spill circumstances.

Bulk density, the mass of a bulk solid occupying a unit volume including interparticle voids, influences oil sorption [1,22]. Specifically, low bulk density favors the formation of capillaries between particles, increasing the available contact area between particles [23]. Although previous studies employed various packing methods such as sphere-shaped mesh bags and tetrahedral oil bags, uniformity of bulk density was not specified which could affect oil sorption [16,24]. Thus, the tetrahedral-shaped mesh from Ismail et al. [16] with

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fixed volume was adapted to ensure a rigid structure and uniform volume, thereby maintaining a constant bulk density throughout sorption while still allowing oil to penetrate into the sorbents [25].

Porosity was found to be higher in rice straw than in rice husk [26]. Studies have also shown that porosity influences sorption more than phase composition since sorption capacity increases along with increasing porosities [14,27]. Thus, rice straw is hypothesized to yield a higher diesel percent sorption than rice husk.

This study aimed to determine and compare the percent sorption of raw rice husk (RRH), raw rice straw (RRS), and sorbent pad for diesel oil. It specifically aims to:

(i) determine the pre-submersion and post-submersion weights (in grams) of the treatment groups (RRH and RRS sorbents) and control group (sorbent pads);

(ii) evaluate the percent sorption (%) of the three sorbents; and

(iii) compare the differences in the mean percent sorption of the treatment groups and control group using one-way ANOVA at $p \leq 0.05$.

Methods. - The rice husks and rice straws were washed, oven-dried, cut, sifted, randomized, and then packed inside a tetrahedral frame wrapped in mesh net (TFMN). After which, the samples were weighed and submerged in pure diesel oil. The control samples, or the sorbent pads, were also subjected to the same packing and submersion procedures. All the sorbents were allowed to drip before getting their post-submersion weights. The percent sorption values were then calculated, compared, and analyzed using One-Way Analysis of Variance (ANOVA) at $p \leq 0.05$. A total of 30 samples, 10 for each type of sorbent, was administered.

Procurement of materials. The RRH and RRS were collected from a rice farm in Guimaras Island (10°43'46.88"N latitude and 122°40'59.594"S longitude) and were verified as *Oryza sativa* L., Rc 418 variety by the Office of Municipal Agriculturist in Buenavista, Guimaras. Meanwhile, the commercial-grade sorbent pads were purchased from YMC Philippines based in Metro Manila. The diesel oil was obtained from a gasoline station in Iloilo City. The tetrahedral frames and mesh net were acquired from local stores in Iloilo City.

Preparation. The RRH and RRS were washed with distilled water and oven-dried at 60°C for 24 hours. The RRS was cut into 9mm similar to that of the RRH, while the RRH remained at its original length. Both of which were hand-sieved through the fine mesh sieves of 3 mm and 5 mm to attain the desired particle size range of 3 to 5 mm.

Randomization. To minimize the biases in this study, the sorbents were subjected to the coning and quartering method by gradually reducing the aggregate to randomly obtain each sample. The oil sorbates were subjected to the "lottery method" to assign them to their respective sample treatment groups with equal chances of being selected.

Packing of Sorbents. All sorbents were packed inside the TFMN as storage for oil retrieval. This was prepared by wrapping a 15x15cm mesh net around the frame, then filled with 3.00 grams of the specific sorbent, and sealed using a plastic heat sealer. The excess fabric was then cut. There were 10 samples for each type of sorbent: RRH, RRS, and sorbent pads, resulting in a total of 30 samples.

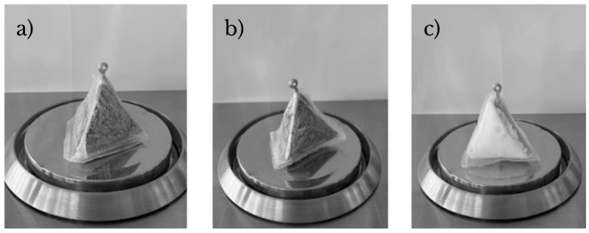

Figure 1. TFMN with a) RRH sample, b) RRS sample, and c) sorbent pad.

Bulk Density. To ensure the uniformity of packing across the sorbents, a constant edge length was set among all TFMN such that the bulk density, when computed, would uniformly equate to 0.28 g/cm^3.

Sorption Test. The packed sorbents were simultaneously submerged in 300 mL of pure diesel oil for 15 minutes and were then allowed to drip for another 15 minutes. To minimize the possible effects of any trapped oil in the TFMN, five (5) replicates of empty TFMN were subjected to the same sorption procedures. After which, each packed sorbent was weighed, followed by subtracting the average weight of the empty TFMNs to obtain the final post-submersion weight of the sorbents only.

Data Analysis. The percent sorption across the sorbents were computed with the formula derived from Razavi et al. (2014):

$$\%\,Sorption = \left(\frac{S_t - S_0}{initial\ oil}\right) x\ 100$$

Where:
S_t = post-submersion weight (in grams) of the sorbent containing the sorbed oil
S_0 = pre-submersion weight (in grams) of the sorbent
initial oil = weight (in grams) of the diesel oil before sorption

Statistical Analysis. There are three independent means to analyze; therefore, one-way ANOVA, along with its applicable inferential non-parametric, parametric, and post-hoc tests, is an applicable statistical test for this study [28]. The computed percent sorption data of each sorbent was inputted in the Jamovi Software (ver. 2.3.17.0) and was subjected to the inferential non-parametric Shapiro-Wilk test to determine the normality of the data. To assess the homogeneity of variances across the normally distributed percent sorption of the sorbents, the parametric Levene's test was utilized.

One-way ANOVA was used to determine the presence of a significant statistical difference across the three mean percent sorption values. The significance level was set at 0.05. Lastly, to make pairwise comparisons between the three means, the Tukey post-hoc test was used to further reveal the specific differences.

Safety Procedure. Health and safety protocols were observed during the conduct of the whole experiment, including wearing of personal protective equipment such as gloves, goggles, mask, and polypropylene laboratory gown. High density polyethylene containers were used for the storage and handling of hazardous chemicals or the diesel oil in accordance with the MSDS. For disposal, excess and contaminated diesel oil and used sorbents were handed to the health and safety officer of Philippine Science High School - Western Visayas Campus.

Results and Discussion. - RRS yielded a mean percent sorption of 4.71 which is significantly higher than RRH of 2.97. Sorbent pads, on the other hand, exhibited the highest mean percent sorption of 7.31. (Table 1)

Table 1. Mean weights of RRH, RRS, and sorbent pads before and after sorption (in grams), and their corresponding mean percent sorption values (%).

Sorbent Type	Before Sorption (g)	After Sorption (g)	Percent Sorption (%)
RRH	3.00±0.0006	10.33±0.38	2.97±0.15
RRS	3.00±0.0002	14.66±0.82	4.71±0.38
Sorbent Pads	3.00±0.0003	21.01±0.66	7.25±0.42

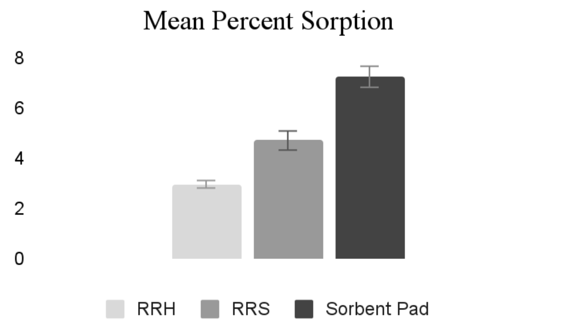

Figure 2. Mean percent sorption of RRH, RRS, and sorbent pad in diesel oil.

The Shapiro-Wilk test revealed a normal distribution among the calculated percent sorption values. The homogeneity of variances in the data was confirmed through the Levene's test. Following the satisfied assumptions, one-way ANOVA indicated a statistically significant difference among the three types of sorbents ($p<0.00001$). The Tukey post-hoc test further showed that sorbent pad has the highest mean percent sorption (7.25%) while RRS (4.71%) exhibited significantly higher sorption than RRH (2.97%).

The oil sorption performances of sorbents can be associated with the hydrophobic-oleophilic properties of the material which in turn were dependent on, porosity, the chemical constituents of the sorbent (i.e. lignocellulosic components), and surface roughness [29]. In addition, bulk density is also recognized as a critical factor in the oil uptake capacities of sorbents [5].

Relating to previous studies, porosity can be accounted for the higher percent sorption of RRS. A study on the physical properties revealed that rice straw has porosity values ranging from 71.21% to 85.28% while rice husk, on the other hand, has values ranging from 63.64% to 73.23% [26]. Porosity is defined as the measure of the volume space occupied by the pores per volume space occupied by the total sample [30]. It enables the incorporation of oil spillages into their matrix in which as porosities of different sorbents increase, the sorption capacities also increase [27,31].

For the filling of pores to occur, the following must be present: (1) the porous structure and (2) the mechanism that will transport the oil into the porous structure, such as the oil adhesion to its surface, thereby promoting capillary movement [32]. RRH and RRS are made up of cellulose, hemicellulose, and lignin with values of 45.8%, 16.9%, 17.2% for RRH, and 42.2%, 23.7%, 13.1% for RRS, respectively [33]. Cellulose and hemicellulose contain hydroxyl (OH) and acid groups that contribute to the samples' hydrophilicity thus limiting their usage for oil cleanup [29,34]. Lignin, on the other hand, is the hydrophobic component of the organic materials. Petroleum components bind to the hydrophobic functional groups of the sorbent for sorption to occur [13]. Previous report has found the higher lignin content of rice husk than rice straw which is in contradiction with the trend observed from the results in this study [33]. Nevertheless, porosity was established to have a stronger influence on sorption performance than phase composition [14]. This may explain the higher sorption of RRS despite having less lignin content than RRH. The high percent sorption of the commercial sorbent pads can be attributed to their high oil recovery ratio once used to their full capacity [20].

A material can be characterized as a sorbent if it allows the sorbate to penetrate into its void through capillary forces or attach to its external surfaces [7]. Focusing on the surface roughness of the samples, this property was suggested to be a part of the adhesion process between the oil and the sorbent's surface [35,36]. Previous report has proven the rough surface of raw rice husk and rice straw [37]. Thus, the oil sorption performance of these sorbents could have also been affected by the aforementioned factor. In light of these, RRH and RRS can be considered as sorbents such that diesel oil was able to attach to their structures, thereby acquiring mean percent sorption values of 2.97% and 4.71% respectively.

During the sorption procedures, similar contact times and dripping times that were previously employed in the investigation involving adsorption and absorption procedures were taken into consideration [38,39]. The 15-minute contact time was adopted in line with the observation that 5 grams of RRH and RRS sorbents yielded the highest sorption values when submerged within the given timeframe [38]. This duration is important in allowing sufficient oil-sorbent contact to ensure that saturation has been reached. Similarly, a 15-minute dripping time was also employed in consideration for real-world applications and also to account for more oil uptake by the sorbent [40].

Varying values of bulk density may influence oil sorption performance, i.e. lower bulk density facilitates the formation of capillaries between particles [23]. This increases the contact area between the particles or porosity, increasing the flow of oil inside the material and also resulting in higher sorption of oil molecules [1]. Taking this into consideration, all the sorbent containers were set with a fixed bulk density of 0.28 g/cm^3. This value was only set to accommodate the sorbents in the container but is not the most optimal value for bulk density. In this light, a tetrahedral-shaped frame served as the rigid structure of the sorbent container, considering that it has the largest surface area among the convex bodies in n-dimensional space [41]. Meanwhile, in attaining a constant volume for the packed sorbents, a mesh net made of nylon was used to wrap around the tetrahedral frame and contain the sorbents for the standardization of the oil sorption test. In the three-dimensional networks of the nylon, the intertwined fibers allowed oil to penetrate into the sorbents [25].

Limitations. Due to limited access to specialized instruments, the determination and in-depth analysis of the mechanisms that specifically occurred during the sorption procedures (i.e. absorption and adsorption) and the characterization of the sorbents using Langmuir isotherm model, SEM analysis, FTIR analysis, etc. were not conducted. Moreover, considering the varying physical shapes of both RRH and RRS, the particle size ranging from 3 mm to 5 mm did not guarantee an equal width and length between samples. Their particle size was solely based on the particles that passed through the 5 mm sieve and remained on top of the 3 mm. Moreover, due to the lack of sieve shakers, the use of hand-sieving procedures did not guarantee uniformity in terms of the rate of strokes and intensity. With regards to the dripping time, the point where no point of further dripping is observed cannot be determined in this study. Lastly, the raw data in four decimal places recorded from the analytical balance were reduced as the statistical software allows the expression of data only in three decimal places. With this, the percent sorption values only represent the rounded-off values obtained from the experiment.

Conclusion. - In evaluating oil sorption capabilities, RRS was found to be a more effective sorbent than RRH, owing to the significantly higher percent sorption of the former in diesel oil. However, RRS is not comparable with the commercially available sorbent pad as the latter displayed prominently higher percent sorption owing to its main design which is intended for industrial oil spills. Nevertheless, RRS can still serve as an alternative sorbent in oil spill emergencies with the significant amount of oil it can sorb. With the utilization of a uniform bulk density through the use of tetrahedral frames, this study provided an improved method for gathering more reliable data for sorption studies.

Recommendations. - To further provide an in-depth analysis of the sorption mechanism and the properties of the sorbents, it is recommended to conduct further analysis (e.g. SEM, FTIR microscopy, etc.) and utilize mathematical models such as the Langmuir isotherm model. To establish uniformity in the particle size, future studies may opt to use machines for uniform cutting and sieving of the materials.

Additionally, in order to assess the full dripping profile of the sorbent, it is recommended to determine and report the value at the "point of no dripping" since the values will not be overestimated or over-reported [8]. A different statistical software is recommended to ensure consistent decimal places from obtaining raw data until analysis. The use of a water-oil system could also be considered to mimic real-life scenarios. In actual oil spill incidents, dry rice husk and rice straw may be contained in a material similar to the mesh net used in the TFMN for easy retrieval after use. In cases where the samples require drying, sun drying, until a constant weight for at least two consecutive days is achieved, may be used as an alternative to oven drying as means of removing any excess moisture from the samples.

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