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# Evaluation of Physicochemical Parameters of Biosorbents Produced from Groundnut Hull Using Microwave Assisted Irradiation Method

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## Abstract

Samples of ground nut hull were converted to biosorbents using microwave assisted method [groundnut hull treated with hydrogen peroxide (HP-GH), groundnut hull treated with distilled water (W-GH) and raw groundnut hull (R-GH)]. The biosorbents were further characterized using physicochemical procedures (pH dependence, bulk density, surface area, ash content, and volatile matter, moisture content). The results show that HP-GH has pH = 8.9, W-GH pH = 8.4 and R-GH pH = 8.5 which is an indication that all the biosorbents have the appropriate pH values for the uptake of cationic species within aqueous systems. Surface area analysis revealed that HP-GH has the largest surface area (74.20 $m^2 \cdot g^{-1}$) while W-GH and R-GH have surface area values of 29.40 $m^2 \cdot g^{-1}$ and 21.40 $m^2 \cdot g^{-1}$ respectively. This suggests that modification of raw groundnut hull biomass with hydrogen peroxide possibly instigated delignification of the biomass which resulted in increased surface area for HP-GH. Results from Bulk density analysis also confirm the data obtained from surface area analysis. Accordingly, R-GH displayed the highest bulk density followed by W-GH with HP-GH showing the least bulk density. The variation in pH values among the biomass used in this study may be explained by the variation in their ash content as well because pH and ash content are positively correlated. Hence, HP-GH with a pH = 8.9 has high ash content (117.31%), W-GH with pH = 8.4 has 97.93% ash content and R-GH with pH = 8.5 has 94.26% ash content. Results from moisture content analysis show that HP-GH (99.95%), W-GH (99.97%) and R-GH (99.89%) may necessitate exposure of the biosorbents to moderate heat before use. The results obtained from this study suggest that modification of ground nut hull with either distilled water or Hydrogen peroxide by means of microwave irradiation improves physicochemical properties which may perhaps increase the adsorption capacity of the biomass.

**Keywords**

Agrowastes, Characterization, Physicochemical, Bulk Density, Surface Area, Volatile Matter, Groundnut Hull, Hydrogen Peroxide

## 1. Introduction

Agricultural wastes are residues from the growing and processing of raw agricultural products such as fruits, vegetables, meat, poultry, dairy products, and crops. They are the non-product outputs from the production and processing of agricultural products that may contain material that can benefit man but whose economic values are less than the cost of collection, transportation, and processing for beneficial use.

Agricultural waste diversity and sustainability issues have become a serious concern lately that have led to huge financial and environmental implications. The lack of proper waste management practices, following the lack of adequate information, and compliance with established protocols has become a challenge too great to be downplayed [1].

Expanding agricultural production has naturally resulted in increased quantities of livestock waste, agricultural crop residues and agro-industrial by-products. There is likely to be a significant increase in agricultural waste globally if developing countries continue to intensify farming systems. It is estimated that about 998 million tons of agricultural waste are produced yearly. Organic wastes can amount to up to 80 percent of the total solid wastes generated in any farm of which manure production can amount up to 5.27 kg/day/1000kg live weight, on a wet weight basis. Agricultural wastes could generate a lot of micro-organisms that cause disease, litter the environment, causes an unpleasant smell and ultimately cause pollution,

The discharge of waste water from industries normally releases effluent containing heavy metals into the environment. Industrial waste water could be categorized into two classes firstly; that generated from electroplating process and secondly; that from rinsing process. In developed countries, heavy metals in waste water are normally removed by advanced technologies such as vacuum evaporation, ion resins, crystallization, membrane technology and solvent extraction [2]. However, these advanced technologies are not readily available in developing countries, [3]. Therefore, it is desired that efficient, simple and pocket friendly removal procedures be developed and used in developing countries. Aside from exhibiting very good biosorption capacity agricultural waste materials are also environmentally friendly and easy to access. Some key advantages of using agricultural wastes as adsorbents includes; easy regeneration and recycling of biosorbents, high efficiency and low-cost [4].

Most agricultural waste materials contain loose and porous structures with functional groups such as carboxyl and hydroxyl that act as viable adsorption sites which in turn enhance their adsorption capacities [5].

Waste management has been a serious challenge, mostly in the developing world, because of the cost implication of treatment procedures [6]. Thus, open dumping and burning have been a common disposal practice of solid waste in most developing countries, and this is truly harmful to human health as well as the environment. Agricultural waste such as peels of fruits and vegetables contribute a large chunk to the solid waste found in most cities and the high demand for groundnut leads to the disposal of large volumes of groundnut hulls found in most environments so there is need to generate adsorbents from them.

Groundnut hulls are a bulky waste generated in large amounts as by-product of peanut (*Arachis hypogea*) processing. Groundnut hulls usually consist of fragmented hulls with variable amounts of whole or broken kernels [7]. In groundnut-producing countries, they are often burned, dumped, or left to deteriorate naturally (Witcombe, 2021). In the recent past environmental concerns have led to an interest in using groundnut shells for a variety of purposes such as; fuel, mulch, carrier for chemicals and fertilizers, bedding for livestock and poultry, pet litter, soil conditioners, etc. [7]. Groundnut hulls are also fed to livestock, particularly ruminants and rabbits, although their high fiber content does not make them suitable for most mono-gastric species. Groundnut hulls are bulky waste generated in large amounts. Groundnut hulls are mostly comprised of fibers such as cellulose (48 wt%), hemicellulose (3 wt%) and lignin (28 wt%). Its chemical composition is essentially composed of silica, iron, oxides, alumina and calcium oxide [7].

Groundnut hull could anchor pathogens associated with gastrointestinal diseases such as diarrhea and dysentery and also create aesthetic nuisance within the environment [8]. In 2020 Ajala and Ali utilized groundnut hulls as a precursor for the preparation of activated charcoal using zinc chloride as an activator. Results from the study show that activated charcoal contains porous structures with adsorption capacities pointedly interrelated with parameters such as iodine value, porosity and surface area [9]. In a similar study Akinola and collaborators investigated the adsorption of Congo red dye from simulated wastewater using Bambara groundnut hulls as adsorbent. The result from the study suggests that Congo red adsorption on Bambara groundnut hull involves chemisorption [10]. In 2021 Shrivastava and associates prepared biosorbents from groundnut hulls and investigated their ability to reduce turbidity of natural and chemical suspensions. Results from the experiments show that biosorbents generated through microwave pyrolysis resulted in substantial lessening in turbidity of up to 96.6% for chemical suspension and 80% for natural suspension [11].

The aim of this study is to synthesizes biosorbents from groundnut hull using microwave irradiation technique and also evaluate the pH, moisture content, bulk density, volatile matter, ash content and surface area of the synthesized biosorbents.

## 2. Materials and Method

### 2.1. Reagents and Materials

All reagents used for this study were of analytical grade (Merck KGaA, Germany).

## 2.2. Sample Preparation/Chemical Activation

The precursor material used for this study (Figure 1) was purchased from local traders at the Swali market, Yenagoa, Bayelsa State, Nigeria. The groundnut pods were manually shelled to separate the nuts and the seed coats from the hulls. The hulls were then washed (12 times) with distilled water and later sun dried for one week. The groundnut hulls were pulverized (blended) using a domestic blender and later separated by size using a sieve (710 μm). Only groundnut hulls that passed through the 710 μm mesh were used for this study. The powdered hull was then stored in an air tight container for further use.

Before any treatment, the sieved biomass was divided into three portions (I, II, and III).

## 2.3. Portion I: Unmodified Groundnut Hull

This portion is left as it is and named raw groundnut hull (R-GH), (Figure 2).

## 2.4. Portion II: Modification of Groundnut Hull Using Distilled Water

Approximately 10 g of the U-GH was weighed into a 250 mL beaker, and 160 mL of distilled water was added. The mixture was subjected to microwave radiation for 5 mins. It was then allowed to cool at room temperature, filtered through a filter paper and washed with distilled water. The residue was dried in an oven for 24 hours at 60˚C, grinded and sieved to particles less than 710 micrometer. The resulting powder was stored in an airtight container labelled D-GH.

Figure 1. Groundnut hull biomass.

Figure 2. Unmodified groundnut hull powder.

### 2.5. Portion III: Modification of Groundnut Hull Using $H_2O_2$ Solution

About, 20 g of untreated groundnut hull powder was weighed into a 250 mL beaker and 170 mL of distilled water was added. The pH of the mixture was adjusted to 10 by adding 0.1M NaOH solution. Approximately 65 mL of 30% $H_2O_2$ was added to the mixture and it was later subjected to microwave radiation for 15 mins and allowed to cool at room temperature, then washed with distilled water, dried in an oven for 24 hours at 60˚C. It was then pulverized and sieved to particles less than 710 μm and labelled HP-GH.

### 2.6. Physicochemical Characterization of Biosorbents

#### 2.6.1. pH Measurement

Approximately 1 g of each sample of the biosorbent namely, raw groundnut hull (R-GH), groundnut hull treated with distilled water (D-GH), Groundnut Hull treated with $H_2O_2$ (HP-GH) and groundnut hull treated with NaOH (Na-GH) is weighed and put in separate 250 mL beaker that contains 100 mL distilled water. This was allowed to boil on a hot plate for 5 mins. The solution is later diluted with 200 mL of distilled water and allowed to cool. The pH of each sample is then measured using a pH meter and recorded accordingly.

#### 2.6.2. Bulk Density Determination

The bulk density of each sample was determined in line with Archimedes' principle. Wherein, a 10 $cm^3$ measuring cylinder is weighed before (empty) and after it was fully packed with each sample and tapped 2 - 3 times, the difference in weight is determined and noted. Bulk density is calculated by using the following equation:

$$\text{Bulk density} = W_2 - W_1 / V \tag{1}$$

where;

$W_1$ = weight of empty measuring cylinder.

$W_2$ = weight of the measuring cylinder with sample.

$V$ = volume of cylinder.

#### 2.6.3. Volatile Matter Determination

In order to determine the volatile matter of the biosorbents 1 g of each sample is weighed and added to a previously weighed empty crucible. Weights of both the crucible and 1 g of each sample are taken. The set up then placed in an oven for 10 mins at 150˚C, after heating, the system is allowed to cool in a desiccator, the volatile matter is calculated using the following expression:

$$\text{Percentage Volatile matter } (\text{Vm}\%) = \frac{W_{vc}(g)}{W_o(g)} \times 100 \tag{2}$$

where,

$W_{vc}$ = Weight of the volatile component [weight of empty crucible ($W_c$)] + weight of sample (1 g).

$W_o$ = Oven dry weight (weight of $W_c$ after oven drying).

#### 2.6.4. Surface Area Determination

The surface area of the biosorbents were determined using the Sear's method in which 0.5 g of each sample was carefully weighed into 250 mL conical flask containing 25 mL of 0.1M HCl (pH 3.50) after which 1 g of NaCl was added to raise the pH to 4, this mixture was then titrated with a standard solution of 0.1M NaOH until a of pH 9 was achieved. The volume needed to increase its pH from 4 to 9 was recorded and used in computing the surface area using Equation (3).

$$\text{Surface area}\left(\text{M}^2/\text{g}\right) = 32V - 25 \tag{3}$$

where, $V$ represents the volume of NaOH used to raise pH from 4 to 9.

#### 2.6.5. Ash Content Determination

The ash content of biosorbents was determined using the procedure previously used by Khalili *et al.*, (2016) in which crucibles containing the samples were preheated to about 500˚C, and cooled in desiccator, after which it was weighed [12]. Approximately 1.0 g of each sample was transferred into the crucible and reweighed The crucible containing the sample were then placed in the furnace and the temperature was allowed to rise 500˚C for 3 hrs and allowed to cool in desiccator to room temperature and weighed. The ash content was calculated using Equation (4):

$$\text{Ash }\% \frac{\text{ash weight}}{\text{oven dry weight}} \times 100 \tag{4}$$

#### 2.6.6. Moisture Content Determination

The moisture content of all biosorbent samples were determined using a procedure previously used by Evbuomwan and coworkers in 2013, where by three empty crucibles were first weighed and 1.0 g of each biosorbent sample was added into the crucible [13]. This was done in triplicate. The crucibles containing samples were oven dried at 110˚C to a constant weight for 3 hours. The samples were then placed in a desiccator to cool and re-weight again. The difference between the initial and final mass of the carbon represents the moisture content. The percentage moisture content is determined using the following expression:

$$\text{Moisture }\% = \left(W_1 - W_2/W_1\right) \times 100 \tag{5}$$

where, $W_1$ = Initial weight of sample (g).

$W_2$ = Final weight of sample after drying (g).

### 2.7. Data Management

Experiments were carried out in triplicate and average values were used for all calculations.

## 3. Results and Discussion

The results of the experimental determination of the physicochemical parameters of the groundnut hull treated with hydrogen peroxide (HP-GH), groundnut Hull treated with distilled water (W-GH) and raw groundnut Hull (R-GH), are shown in **Figures 3-8**.

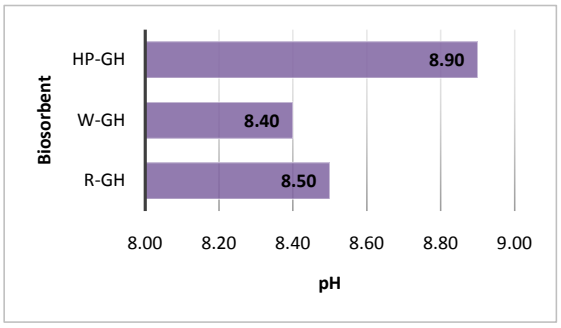


**Figure 3.** pH values for biosorbents.

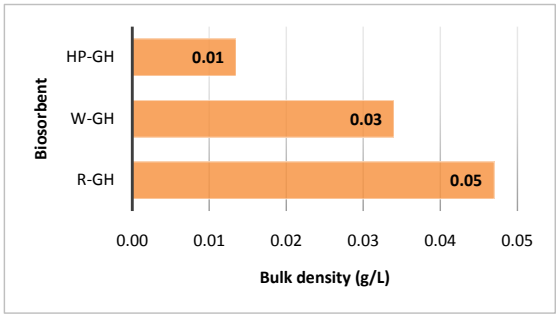


**Figure 4.** Bulk Density of biosorbents.

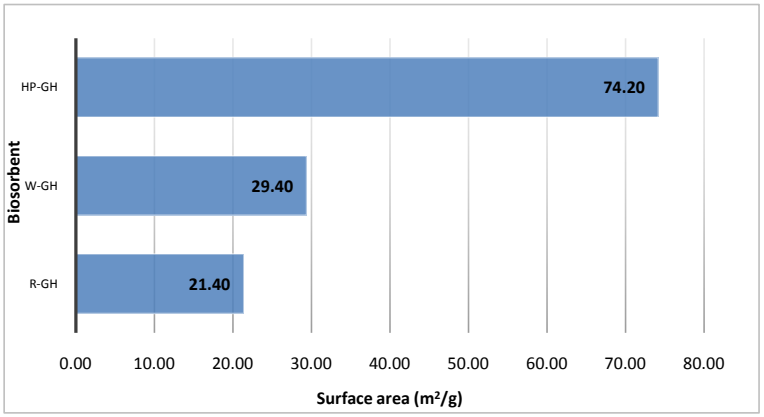


**Figure 5.** Surface Area values for biosorbents.

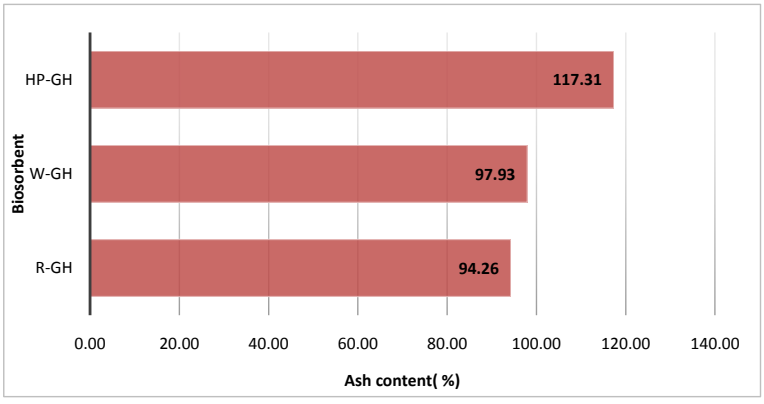


**Figure 6.** Ash Content of biosorbents.

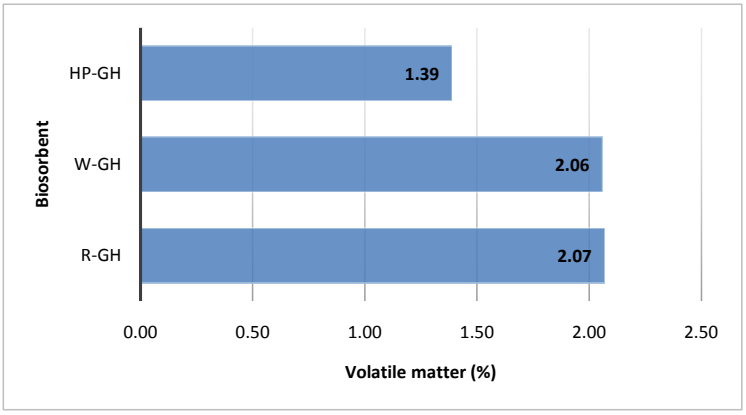


**Figure 7.** Percentage Volatile Matter of biosorbents.

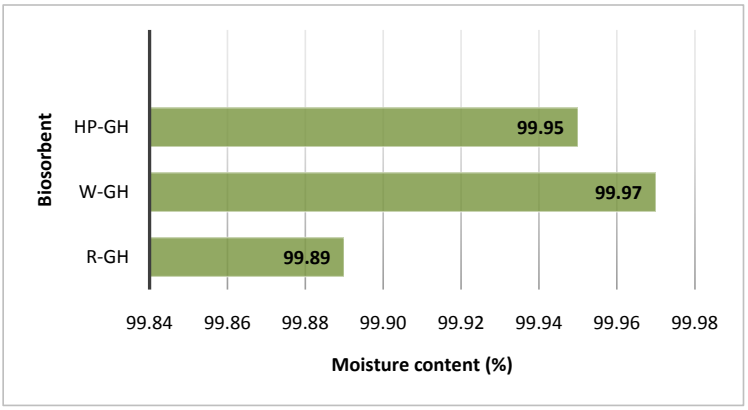


**Figure 8.** Percentage moisture content of biosorbents.

### 3.1. pH Dependence

The pH of an adsorbent has a significant effect on its ability to pick up adsorbates during adsorption processes, this is due to the fact that pH controls the ionic state of functional groups that exist as active adsorption sites on the surface of the adsorbent [14] [15]. In 2018 Manirethan and colleagues posited that as pH increases, positive partial charges on the surface of an adsorbent decrease [16]. Results from a previous report suggested that the maximum adsorption of metal ions by most activated carbonaceous adsorbents occur between pH 6-9 [17] [18]. Based on the aforementioned one can without difficulty deduce from **Figure 3**, that all three (3) biosorbents used in this study have pH values that are above 8.0, which means they all possess negatively charged surfaces which could be exceptionally good for the uptake of positively charged species.

### 3.2. Bulk Density/Surface Area

Bulk density describes the mass of an adsorbent in a specific volume [19]. The bulk densities of HP-GH, W-GH and R-GH were calculated as 0.01, 0.03 and 0.05 $g{\cdot}L^{-1}$ respectively (see **Figure 4**). From the results shown in **Figure 4**, R-GH exhibited the highest bulk density followed by W-GH and HP-GH showed the least bulk density. **Table 1** is a comparison of bulk densities of biosorbents used

**Table 1.** A comparison of bulk densities HP-GH, W-GH and R-GH with that of similar biosorbents.

| Biosorbent | Bulk density ($g \cdot L^{-1}$) | Reference |
|---|---|---|
| HP-GH | 0.01 | This study |
| W-GH | 0.03 | This study |
| R-GH | 0.05 | This study |
| HP-WMR | 0.41 | [20] |
| DW-WMR | 0.46 | [20] |
| U-WMR | 0.61 | [20] |

[Untreated Watermelon Rind (U-WMR), Watermelon Rind modified with distilled water(D-WMR), Watermelon Rind modified with Hydrogen Peroxide (HP-WMR)].

in this study and that of similar biosorbents in a previous study, the result reveals that HP-GH, W-GH and R-GH exhibited lower bulk densities than what was obtained for similar biosorbents.

Surface area available for adsorption per gram of adsorbent is known as specific surface area and any increase in surface area of an adsorbent increases the adsorption capacity of the adsorbent [21]. Thus, specific surface area is a very important parameter in adsorption studies because it is used to evaluate the adsorption capacity of adsorbents. The result from surface area analysis shown in **Figure 5**, illustrate that HP-GH has the largest surface area (74.20 $m^2 \cdot g^{-1}$) while W-GH and R-GH have surface area values of 29.40 $m^2 \cdot g^{-1}$ and 21.40 $m^2 \cdot g^{-1}$ respectively. This suggests that modification of raw groundnut hull biomass with hydrogen peroxide resulted to delignification of the biomass and this in turn increased the surface area of the biomass [22]. Moreso, the observed differences in surface area among the biomass investigated in this study might be attributed to the nature of the precursor biomass, irradiation temperature and the kind of modification [22].

### 3.3. Ash Content/Volatile Matter

The total amount of minerals present in a material is known as ash content; it is a measure of the quantity of inorganic components found in the material. Ash content data obtained in this study is displayed in a chart format (**Figure 6**). This result shows a very high amount of ash in all the biosorbents and this could be a cause for concern because ash content has a tendency of reducing the overall efficacy of adsorbents in terms of re-use [23].

Volatile matter is a parameter that describes the number of materials degraded from an adsorbent within a certain temperature range. In 2012 a study by Budianto *et al.* [24] reported that maximum volatile matter allowed for activated carbon is 25%. **Figure 7** shows the volatile matter content of HP-GH, W-GH and R-GH as 1.39, 2.06 and 2.06% respectively which implies that much volatile matter was released from the biosorbents due to microwave irradiation and hydrogen peroxide treatment. Hence, all three biosorbents met the requirement of

volatile matter.

### 3.4. Moisture Content

Percentage moisture in an adsorbent can be described as the quantity of liquid especially water present within an adsorbent mostly in trace amounts. Scientific reports have shown that moisture content increases linearly with Bulk density thus, the existence of moisture in an adsorbent is conventionally not good for normal applications [25] [26]. Therefore, high moisture content does not support adsorption while low moisture content enhances adsorption capacity of adsorbents.

Observing **Figure 8**, one can construe that all three (3) biosorbents have high moisture content (>90%) therefore, it will be desirable to subject the biosorbents to slight heat for some time before they are used as adsorbents, as this will lessen their moisture content and improve their adsorption capacity [27].

## 4. Conclusions

Three biosorbents (HP-GH, W-GH, R-GH) were prepared from groundnut hull using microwave irradiation assisted method. The biosorbents were further characterized using physicochemical procedures.

Experimental results show that HP-GH has a pH of 8.9, W-GH has a pH of 8.4 and R-GH has a pH of 8.5 which is an indication that all the biosorbents have the appropriate pH values for the uptake of cationic species within aqueous systems.

Surface area analysis of the biosorbents reveals that HP-GH has the largest surface area (74.20 $m^2 \cdot g^{-1}$) while W-GH and R-GH have surface area values of 29.40 $m^2 \cdot g^{-1}$ and 21.40 $m^2 \cdot g^{-1}$ respectively. This proposes that modification of raw groundnut hull biomass with hydrogen peroxide caused delignification of the biomass which resulted in increased surface area for HP-GH. Results from Bulk density analysis also validate the data obtained from surface area analysis because bulk density is inversely proportional to the surface area. Accordingly, R-GH exhibited the highest bulk density followed by W-GH and HP-GH exhibited the least bulk density.

The variation in pH values among the biomass used in this study may be explained by the variation in their ash content as well because pH and ash content are positively correlated. Hence, HP-GH with a pH = 8.9 has high ash content (117.31%), W-GH with pH = 8.4 has 97.93% ash content and R-GH with pH = 8.5 has 94.26% ash content. Results from moisture content analysis show that HP-GH (99.95%), W-GH (99.97%) and R-GH (99.89%), this is a clear indication of possible challenges with respect to re-use. Therefore, it is necessary to expose the biosorbents to heat before use.

The results obtained from this study suggest that modification of groundnut hull with either distilled water or Hydrogen peroxide by means of microwave irradiation, improves physicochemical properties which may perhaps increase the

adsorption capacity of the biomass. Thus, the result shows that all the three synthesized biosorbents have the features of a good adsorbent.

## Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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