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Preparation of Rice Husk Bio-Adsorbent and its Application for the Removal of Methylene Blue from Water: An Experimental Study

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ABSTRACT

Water pollution caused by coloured organic compounds is an important environmental concern because dyes released from different industrial activities can affect water quality and aquatic ecosystems. Conventional adsorbents such as activated carbon are effective but may involve relatively high preparation and operating costs. Agricultural wastes provide an alternative source of low-cost and renewable adsorbent materials. Rice husk, an agricultural by-product of rice milling, contains lignocellulosic components and silica and has been investigated as a potential bio-based adsorbent for the removal of pollutants from water. The present experimental study focuses on the preparation of a simple rice husk bio-adsorbent and its application for the removal of methylene blue from aqueous solution. The study involved a simple laboratory experiment that can be undertaken by undergraduate students. The rice husk was collected, washed with water, dried, powdered and sieved to acquire a somewhat uniform adsorbent. A known concentration of methylene blue solution was then exposed to a fixed amount of the obtained rice husk bio-adsorbent. Contact time was chosen as the sole variable and samples were subjected to colorimetric analysis at various time intervals. Residual methylene blue concentration was calculated using a pre-prepared calibration curve while dye removal efficiency was estimated using the initial and final concentrations of methylene blue. This study illustrates a simple way through which an agricultural waste product can be made useful as an adsorbent for water purification process. In addition, the study gives undergraduate students exposure on how agricultural waste products can be made valuable as well as exposure on adsorption, solution preparation, colorimetric analysis and environmental chemistry among others. This experiment has significance in sustainable development because it integrates both agricultural waste utilization and a cheap method of water pollution management.

***Keywords**: Rice Husk, Bio-Adsorbent, Methylene Blue, Adsorption, Agricultural Waste, Wastewater Treatment, Sustainable Development, Colorimetry.*

Introduction

Water is one of the most important natural resources required for human life, agriculture, industry and ecosystems. Increasing industrialization and urbanization have resulted in the discharge of different types of pollutants into water bodies. Among these pollutants, synthetic dyes are important because even relatively small quantities can impart intense colour to water and may interfere with light penetration and aquatic processes. Therefore, the removal of coloured pollutants from contaminated water is an important area of environmental research.

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Several techniques are available for wastewater treatment, including coagulation, filtration, oxidation, membrane processes and adsorption. Among these methods, adsorption is widely investigated because of its simplicity and effectiveness. Activated carbon is one of the most commonly used adsorbents, but the cost associated with its preparation and use encourages researchers to investigate inexpensive alternative materials.

Agricultural residues are particularly attractive because they are renewable, widely available and can often be converted into useful products. Materials such as rice husk, rice straw, sawdust, coconut shell, banana peel and other plant residues have been investigated as biosorbents. Reviews of rice-husk-based adsorbents report their potential for removing different pollutants, including dyes and other organic contaminants, from water.

Rice husk is the outer covering removed during rice milling. Instead of treating this material only as agricultural waste, it can be considered a potential resource for environmental applications. The rice husk is a lignocellulosic material containing silica, and the surface of the rice husk contains functional groups capable of interacting with soluble species. High abundance, availability, and inexpensive nature of the rice husk make it a promising adsorbent for further investigations.

In this experiment, methylene blue is chosen as the model colored contaminant. The choice is made due to the ability of the methylene blue to be easily measured with the colorimeter due to its colored nature. Such an approach makes the experiment quite suitable for performing the experiment in undergraduate laboratory.

Thus, the primary goal of the current experiment is to synthesize the bio-adsorbent from rice husk without any chemical activation and investigation of its ability to remove methylene blue from aqueous solution.

Need and Significance of the Study

Agricultural operations produce significant amounts of residues. The use of such residues for preparing useful products is in line with the concept of waste reduction and recycling. The rice husk is a material that is particularly suited for an undergraduate research since it can be converted to useful materials via simple physical methods like washing, drying, grinding, and sieving.

The importance of this particular experiment can be outlined as follows:

- An example of converting an agricultural waste product into an environmentally valuable material is provided by the experiment.
- The concept of adsorption is introduced to the students.
- The students learn how to prepare standard solutions and dilutions.
- The skills of performing colorimetry are developed among the students.
- The application of chemistry to environmental issues is shown.
- The sustainable utilization of waste is promoted among the students.
- The experiment involves relatively simple laboratory equipment.
- The effect of contact time on adsorption can be studied.

Studies have proved that rice husk is one of the most inexpensive and sustainable adsorbents with possible use in pollutants' removal process. However, studies show that chemical modification might increase the efficiency of adsorption process. This study deliberately does not employ chemical activation to make the experiment suitable for undergraduate students.



Objectives of the Study

The study was conducted with the following three objectives:

- To prepare a bio-adsorbent from rice husk agricultural waste.
- To investigate the effectiveness of rice husk bio-adsorbent for removing methylene blue from water.
- To study the effect of contact time on the dye-removal efficiency of rice husk bio-adsorbent.

Hypothesis

The study was based on the following hypothesis:

H_0: Contact time does not significantly affect the removal of methylene blue by the prepared rice husk bio-adsorbent.

H_1: Contact time significantly affects the removal of methylene blue by the prepared rice husk bio-adsorbent.

For a simple undergraduate investigation, the hypothesis can be evaluated by comparing the percentage removal obtained at different contact times.

Materials and Methods

- **Materials Required**

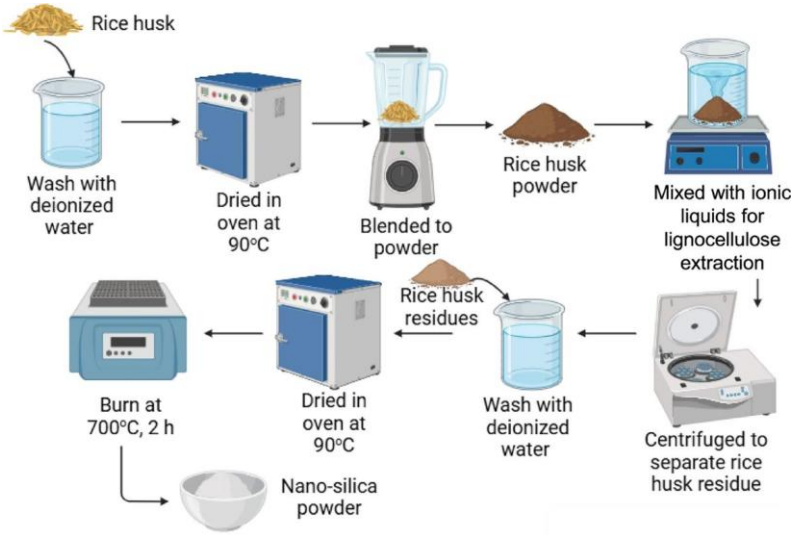
  The following materials and equipment are required:

  - Rice husk
  - Methylene blue
  - Distilled water
  - Beakers
  - Conical flasks
  - Measuring cylinders
  - Volumetric flasks
  - Pipettes
  - Funnel
  - Filter paper

- Glass rod
- Digital balance
- Mortar and pestle or laboratory grinder
- Sieve
- Hot-air oven
- Shaker, if available
- Colorimeter
- Cuvettes
- Stopwatch
- Labels and marker

The experiment does not require chemical activation of the rice husk.





- **Preparation of Rice Husk Bio-Adsorbent**

Rice husks were collected from a rice milling factory and examined to eliminate visible foreign materials.

Rice husk was repeatedly washed using tap water to eliminate dust particles and other impurities attached to it. It was then washed using distilled water. The washed husk was spread out on a clean surface and allowed to dry. This may be done either by exposure to sunlight or drying in a laboratory oven at an appropriate temperature till constant dryness is achieved. Laboratory drying process gives more control in preparing the material.

Dried rice husks were pulverized using mortar and pestle or appropriate laboratory pulverizing equipment. Powdered samples were sieved to give uniformly-sized powder particles. The prepared material was kept in a clean and labelled container.

The preparation process can thus be summarized as follows:

Rice husk → Washing → Drying → Pulverizing → Sieving → Rice husk bio-adsorbent



- **Preparation of Methylene Blue Solution**

A stock solution of methylene blue was prepared using distilled water according to the concentration selected for the experiment.

A working solution was prepared from the stock solution by appropriate dilution. All solutions were prepared using volumetric glassware to improve accuracy.

For the colorimetric analysis, a series of standard methylene blue solutions of known concentrations should preferably be prepared. Their absorbance/colorimeter readings are recorded to construct a calibration curve.

The calibration curve should be prepared before analysing the experimental samples.

- **Colorimeter Calibration**

The colorimeter was switched on and allowed to stabilize according to the manufacturer's instructions.

Distilled water was used as the blank. The appropriate filter/wavelength for methylene blue was selected according to the instrument and laboratory protocol. Methylene blue is commonly monitored in the region around 660–665 nm using spectrophotometric measurements; when a filter colorimeter is used, the closest appropriate filter available on the instrument should be selected.

The standard methylene blue solutions were measured and their readings were recorded.

A graph was plotted with:

  - **X-axis:** Concentration of methylene blue
  - **Y-axis:** Colorimeter reading/absorbance

The resulting calibration relationship was used to determine the concentration of methylene blue remaining in the treated samples.

- **Adsorption Experiment**

A fixed volume of methylene blue working solution was transferred into a series of labelled conical flasks.

Fixed amount of processed rice husk bi-adsorbent was put into each flask. For instance, an experimental setup might have 1.0 g of rice husk bi-adsorbent with 100 mL of methylene blue solution, if the dose is appropriate for a chosen concentration in the laboratory.

Flasks were shaken at a fixed rate. Contact time was varied while all other experimental conditions were kept constant.

Suggested contact times were:

- 15 minutes
- 30 minutes
- 45 minutes
- 60 minutes
- 90 minutes

At the end of each contact period, the mixture was filtered to separate the rice husk adsorbent from the solution.

The filtrate was collected in labelled containers and its colorimeter reading was measured.

For improved reliability, each experimental condition should preferably be performed in triplicate and the mean value should be reported.

Experimental Variables

To make the experiment simple, only one major independent variable was changed.

Parameter	Experimental Condition
Adsorbent	Rice husk
Pollutant	Methylene blue
Adsorbent dose	Fixed
Solution volume	Fixed
Initial dye concentration	Fixed
Temperature	Approximately constant
Mixing condition	Constant
Contact time	15, 30, 45, 60 and 90 min
Analytical method	Colorimetry

Keeping the other conditions constant makes it easier for students to understand the effect of contact time.

Observation Table

The following table can be used by the students during the experiment.

For the following illustrative calculation, the initial methylene blue concentration (C_0) is taken as 10 mg/L. The rice husk adsorbent dose is 1.0 g in 100 mL solution, with all other experimental conditions maintained constant. The concentration values below are illustrative/sample values and must be replaced by actual laboratory observations before final submission.

Contact Time (min)	Trial 1 C_t (mg/L)	Trial 2 C_t (mg/L)	Trial 3 C_t (mg/L)	Mean C_t (mg/L)	Removal (%)
15	7.8	7.6	7.7	7.70	23.0
30	6.3	6.1	6.2	6.20	38.0
45	5.1	4.9	5.0	5.00	50.0
60	4.2	4.0	4.1	4.10	59.0
90	3.8	3.6	3.7	3.70	63.0

Calculation

The percentage removal of methylene blue can be calculated using:

$$\textbf{Removal (\%)} = [(C_0 - C_t) / C_0] \times 100$$

Where:

- C_0 = initial concentration of methylene blue
- C_t = concentration of methylene blue after contact time *t*

For example, if the calibration curve provides the concentration remaining in the solution after treatment, that value can be used as C_t.

The adsorption capacity can also be calculated if required:

$$q_t = [(C_0 - C_t)V] / m$$

Where:

- q_t = amount of dye adsorbed per unit mass of adsorbent
- C_0 = initial concentration
- C_t = concentration after contact time
- **V** = volume of solution
- **m** = mass of adsorbent

Mean C_t = (Trial 1 + Trial 2 + Trial 3) / 3.

Removal (%) = $[(C_0 - C_t) / C_0] \times 100$.

15 min: Mean C_t = 7.70 mg/L; Removal (%) = [(10.00 − 7.70)/10.00] × 100 = 23.0%.

30 min: Mean C_t = 6.20 mg/L; Removal (%) = [(10.00 − 6.20)/10.00] × 100 = 38.0%.

45 min: Mean C_t = 5.00 mg/L; Removal (%) = [(10.00 − 5.00)/10.00] × 100 = 50.0%.

60 min: Mean C_t = 4.10 mg/L; Removal (%) = [(10.00 − 4.10)/10.00] × 100 = 59.0%.

90 min: Mean C_t = 3.70 mg/L; Removal (%) = [(10.00 − 3.70)/10.00] × 100 = 63.0%.

Graphical Representation

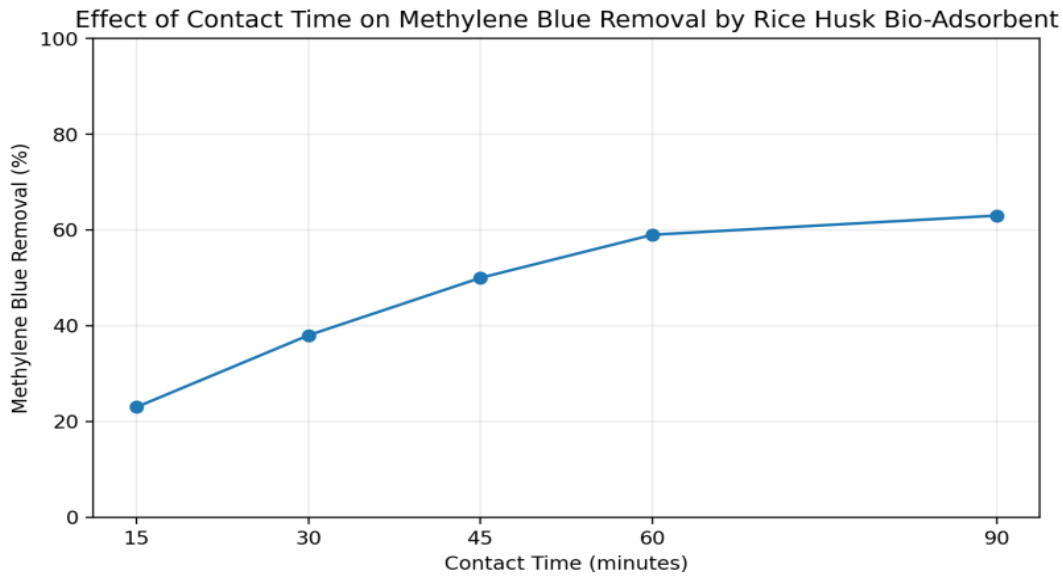


Figure 1: Effect of Contact Time on Percentage Removal of Methylene Blue by Rice Husk Bio-Adsorbent (Illustrative/Sample Data)

The rice husk bio-adsorbent displayed significant capacity for removal of methylene blue from aqueous solution in the set conditions of experimentation.

- Removal of methylene blue increased gradually with increasing contact time for the illustrative dataset, rising from 23.0% after 15 minutes to 63.0% after 90 minutes.
- Maximum removal in the illustrative data was achieved after 90 minutes.
- Rate of increase decreased with increasing contact time, reflecting an approach to adsorption equilibrium.

- This is in line with gradual filling of available adsorption sites with increasing contact time.
- The experiment illustrates the use potential of untreated, physically produced rice husk as a cost-effective agricultural waste adsorbent for aqueous solution of dyes.
- The results pertain to lab-prepared methylene blue solution only and cannot be directly applied to industrial wastewater treatment.

Result

As seen in the illustrative figures below, there is a positive relationship between the amount of methylene blue removal and the increase in contact time. The average percent removal is 23.0% at 15 minutes and 63.0% at 90 minutes. The trend begins to level off as contact time increases. The maximum percent removal is 63.0% at 90 minutes.

Discussion

The adsorption of methylene blue using rice husk can be considered as an interaction between the dissolved dye molecules and the surface of the prepared waste agricultural material. Adsorbents obtained from plants have various components on their surface which can participate in the interaction with dissolved substances.

In the beginning of the adsorption experiment, one can expect the surface sites available for interactions to be quite numerous. Thus, the interaction of dye molecules with these sites will take place and their concentration in solution will decrease. With the increase of contact time, more and more sites can become occupied. Thus, the rate of removal will depend on the increase of contact time.

The exact tendency revealed in the current experiment can be obtained according to the data of students' experiments. If the percentage of removal increases at the beginning of time intervals and then remains constant, one can say that adsorption approaches equilibrium.

Several studies have shown that rice husk is a good adsorbent in terms of cost for different types of pollutants in the scientific literature. The conclusion of the review of adsorption studies based on rice husk has indicated that rice husk may be used to remove different types of pollutants such as dyes. However, pretreatment might affect adsorption effectiveness.

This study differs from other advanced researches by using non-prepared rice husk. The study makes the experiment better for undergraduate students.

The results cannot be regarded as a treatment of real industrial wastewater. The use of methylene blue solution is done to simulate wastewater pollution in the laboratory. Real wastewater consists of many different substances that may inhibit adsorption, including salts, organic matter, and other pollutants.

Environmental and Sustainable Development Significance

The current work deals with three critical issues in the environment: agricultural waste management, water pollution and sustainable use of resources.

Rice husk is produced as the by-product of rice cultivation. Instead of perceiving it as the waste, one may analyze its potential use in environmental applications. It corresponds to the idea of waste valorization, which implies transformation of the waste material into something more valuable.

Agricultural residue as adsorbent has been proposed as a method to solve waste-management and water treatment problems at once.

Also, the project involves some sustainable aspects:

- The use of renewable agricultural waste.
- The reduction in use of expensive adsorbents.
- Implementation of simple treatment techniques.
- Circular economy ideas at the laboratory scale.
- The combination of chemistry with environmental protection.
- The development of practical research skills of the students.

So, the project is in accordance with the topic of the conference “Science, Technology, Environment, Health & Sustainable Future”.

Advantages of the Proposed Method

This proposed experimental technique possesses a number of strengths:

- The raw material rice husks is waste product from agriculture and hence cost effective.
- The preparation process of adsorbent is easy to perform.
- There is no need of any chemical activation process.
- Common laboratory apparatus is sufficient for this experiment.
- Colorimeter can be used for analysis purpose.
- The technique is appropriate for practical research at undergraduate level.
- Experiment shows the significance of chemistry in environment.
- The research involves waste recycling.
- The experiment can be completed within laboratory time frame.

Limitations of the Study

Limitations of the Study

First, the test has been carried out on laboratory prepared solution of methylene blue instead of real wastewater. So, the result obtained cannot give a direct indication of the efficiency of the wastewater treatment process.

Second, raw rice husk may exhibit poor adsorption capability compared to chemically treated adsorbent. Literature reveals that pretreatment or modification may affect the adsorption capability of rice husk.

Third, in the current study only the contact time is taken into consideration. There could be other factors such as pH, adsorbent dose, concentration of dye, temperature and particle size affecting the process.

Fourth, laboratory scale experimentation has been carried out here. Further research work will be necessary to determine the other aspects such as regeneration and reuse.

Future Scope

The current research may be further pursued by undergraduate students or future researchers in a number of ways.

- The effects of varying amounts of the adsorbent may be examined.
- The effect of pH may be studied.
- Variations in particle size of rice husk may be considered.
- Variations in the initial concentrations of methylene blue may be taken into account.
- The effects of chemical or thermal treatments of the rice husk may be compared with those of the untreated rice husk.
- The reusability of the adsorbent may be analyzed.
- Other dyes may be studied.
- Real wastewater samples may be studied under proper safety measures and analytical techniques.
- Adsorption isotherms and kinetic models may be examined in more detail.
- The economic viability of water treatment using rice husks may be studied.

Such researches may help to establish if adsorbents using rice husks can be developed for practical purposes.

Conclusion

The current work offers an easy and eco-relevant way of transforming rice husk waste into bio-adsorbent for methylene blue removal from aqueous solutions. The preparation of the adsorbent is based on a sequence of physical manipulations, such as washing, drying, grinding, and sieving, thus making it relevant for B.Sc. Third-Year students.

The use of colorimeter allows students to measure easily the concentration of methylene blue before and after the process and learn the essence of the adsorption process as well as its dependence on the contact time at a constant level of other experimental parameters.

It can be considered as a good combination of basic chemistry, environmental science, and sustainable development, showing how agricultural waste can be researched as a possible adsorbent. According to the existing literature, the rice husk can be used as a cheap and renewable adsorbent; however, there is also the information about possible improvement of the material properties.

Thus, the proposed experiment can be considered as a good way of educating B.Sc. third-year students in experimental environmental chemistry. The effectiveness of the rice husk bio-adsorbent must be confirmed by the results obtained with the help of colorimeter.

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Author Contribution

The student researchers contributed to the preparation of rice husk bio-adsorbent, laboratory experimentation, colorimetric analysis, recording of observations and interpretation of results. The supervising faculty member guided the experimental design, laboratory procedures, data analysis and preparation of the manuscript.

Declaration

The study is intended as a laboratory-scale undergraduate experimental investigation. The numerical results presented in the final conference manuscript should be based exclusively on the observations obtained during the actual experiment.

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