Fixed Bed Column Study for Removal of Basic Blue 90 Dye from Simulated Wastewater Using Hybrid Modified Rice Husk and Carbonized Palm Shell Adsorbents

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الملخص

يعد وجود جزيئات الأصباغ في القنوات المائية إنذارًا ناشئًا لعالم البيئة. ينبغي استكشاف طريقة صديقة للبيئة ومستدامة ذاتياً لمعالجة هذه المشكلة. لذلك كان الهدف من هذا العمل هو دراسة امتزاز الصبغة الزرقاء الأساسية (90) من مياه الصرف الصناعي باستخدام الممتز الهجين المعدل من قشر الأرز المخلوط (BRH) وقشور النخيل المتفحمة (CPS) في عمود الامتزاز احادي الطبقات ومتعدد الطبقات. لهذا الغرض، تم تحضير ستة أنواع من الممتزات لاستخدامها لإزالة الصبغة وهي مادة ماصة واحدة من قشر الأرز غير المخلوط بنسبة (100٪ BRH)، واحدة من غلاف نواة النخيل المتفحمة غير مخلوطة بنسبة (100٪ CPS) ، وأربعة مواد ماصة هجينة محضرة بخلط قشر الارزالمخلوط وقشور النخيل المتفحمة بنسب خلط 20 ٪ ،40 ٪ ، 60٪ ، 80٪ بالوزن من قشور الارز المخلوط . كل مادة ماصة تم توصيفها بشكل فردي عن طريق التحليل العنصري ومساحة سطح والمجهر الإلكتروني لمسح الانبعاثات الميدانية، تم تقييم أداء عمود الامتزاز لكل مادة ماصة عن طريق كفاءة الإزالة بالتحليل البياني للمساحة تحت المنحنى. أظهرت النتائج بأنه لكل من عمود الامتزاز المفرد والمتعدد الطبقات، تم امتصاص الصبغة بقوة بنسبة 100٪ باستخدام المادة المازة المكونة من قشور الارز

المخلوط بنسبة 100٪ مع كفاءة إزالة بلغت 92.92 ٪، و97.17 ٪، بأقل مساحة تحت الرسم البياني بلغت 575.95، و571.05 على التوالى. كخلاصة ،استخدام قشر الأرز غير المخلوط كمادة ماصة لإزالة الصبغة الزرقاء 90 أظهر أقصى كفاءة إزالة مع أقل مساحة تحت الرسم المنحنى، وخلط قشر الأرز مع قشور النخيل المتفحمة كمادة ماصة هجينة لم يكشف عن أي تأثير مفيد لزيادة كفاءة الإزالة للصبغة المدروسة.

Abstract

The presence of dye molecules in water channels is an emerging alarm to an environmental scientist. An environmental friendly and self-sustainable treatment method should be explored to address this problem. Therefore, the aim of this work was to investigate the adsorption of Basic blue 90 dye (BB-D) from synthetic waste water using hybrid modified adsorbent from blended rice husk (BRH) and carbonized palm shell (CPS) in single and multi- layered beds adsorption column. For this purpose, six types of adsorbents were prepared and used for dye removal which are one unmixed rice husk (100% BRH) adsorbent; one unmixed carbonized palm kernel shell (100% CPS) adsorbent; and four hybrid adsorbents prepared by mixing (BRH) and (CPS) with mixing ratios 20%,40%,60%,80% wt of BRH .Each adsorbent was characterized individually by elemental analysis, BET surface area, and Field Emission Scanning Electron Microscope (FESEM). Evaluating the performance of the column for each adsorbent was carried out by means of removal efficiency and area under the graph analysis. The results showed that, for both single and multi – layered bed column the adsorption of dye was strongly adsorbed by 100% RH (BRH)) with removal efficiency of 92.92%, and 97.17%, with least area under the graph of 575.95, and 571.05, respectively. As a conclusion, using unmixed rice husk as adsorbent for (BB-D) dye removal showed maximum removal efficiency with least area under the graph and mixing

of rice husk with CPS as a hybrid adsorbent did not reveal any beneficial effect for increasing the removal efficiency of studied dye.

Index Terms: Removal , Basic Blue 90 dye,wastewater,rice husk, carbonized palm shell .

1. Introduction

Dyeing industry is one of the largest water consuming industries (K. Siddique et al. 2017). At present, there are more than 10,000 commercial dyes and the annual production of these dyes is estimated to be $7x10^5$ tons (Islam et al.2018). It is reported that 10–15% of the used dyes enter the environment through wastes (Hai et al.2007; Husain,2006). The main consumers of dyes are textile, dyeing, paper and pulp, tannery and paint industries, and hence the effluents of these industries as well as those from plants manufacturing dyes tend to contain dyes in sufficient quantities. Dyes are considered an objectionable type of pollutant because they can endanger human health. It can enter the body by oral ingestion and inhalation and cause skin and eye irritation (Christie,2007; Rai et al.2005).Due to the environmental and health impact of these dyes in wastewater effluents, many technologies have been developed for purification and treatment of wastewater consists of various chemical, physical and biological. These including chemical precipitation, solvent extraction, oxidation, reduction, dialysis /electro dialysis, electrolytic extraction, reverse osmosis, ion-exchange, evaporation, cementation, dilution, adsorption, filtration, floatation, air stripping, stream stripping, flocculation, sedimentation and soil flushing/ washing chelation processes (Burguera et al.2000, Vital RK et al.2016). It is important to evaluate the technology's selection based on several factors such as available space for construction of treatment facilities, ability of process equipment, limitation of waste disposal, desired final water quality, and costs of capital and operating. Among all the process, adsorption is found to be one of the most suitable techniques to remove pollutants from wastewater. It is highly preferred due to its high reliability, energy efficiency, design flexibility, technological maturity and the ability to regenerate the exhausted adsorbent (Crini et al. 2008). Apart from removing many types of pollutants, adsorption also has wide application in water pollution control (Tang et al.2017). Activated carbon has been identified as the most widely used adsorbent for adsorption process to remove various types of dyes. This is due to its higher surface area and porous structure, which then lead to higher adsorption capacity. Despite of these advantages, the manufacturing cost of adsorption process is expensive due to high cost of activated carbon and regeneration process. In an attempt to address the cost problems associated with commercial adsorbents; the use of abundant, locally available, low cost adsorbents derived from agricultural wastes which are oil palm shell and rice husk are proposed in this study. However, the application of untreated agricultural wastes material as adsorbents can also bring several problems such as low adsorption capacity (Gaballah et al.1997). Therefore, these materials need to be modified or treated before being applied for the decontamination of dyes. Most of the previous researchers focused on the removal of dyes by using agriculture waste materials by single bed column adsorption such as, Methylene Blue (IAW et al.2008), Malachite Green (Ahmad MA et al.2011), Rhodamine B (Panneerselvam et al.2012) and Acid Orange 52 (AO 52) (Jumasiah et al.2005). In particular, there is a need to investigate the effect of the number of beds via multi-bed column adsorption for the removal of dyes components from simulated wastewater. This research was conducted to examine the potential to generate a synthetic adsorbent from CPS and BRH to be used for the removal of Basic blue 90 dye (BB-D) from wastewater. For this purpose, single and multi-beds adsorption experiments were conducted to measure the removal percentages of Basic blue 90 dye (BB-D) using the modified hybrid adsorbents.

2. Methodology

2.1 Preparation of modified hybrid Adsorbents

Raw rice husk was obtained from *PadiBerasNasionalBerhad* (BERNAS) in Sekinchan, Selangor, Malaysia and Palm kernel shells were collected from the Palm Oil Mill Technology Center (POMTEC), Labu, Negeri Sembilan, Malaysia. The Raw Rice Husk and Palm kernel shells were washed with distilled water and filtered prior to drying at the temperature range of 105 – 110°C.The raw rice husk was then blended and sieved to 150 – 250 μm size using Retsch mechanical sieve shaker and the final obtained adsorbent product was named as blended rice husk (BRH). The sample was stored in plastic bottles and used for the reference of experiments for each dye. On the other hand, the raw palm kernel was carbonized at 600°C in a furnace for 5 hours. The carbonized palm shells were then underwent size reduction by grinding using the waring commercial laboratory blender and sieved to the range of 150 to 250 µm. At this point, grinded carbonized palm shell was termed as (CPS).

For each mode of adsorption study six hybrid adsorbents with total amount of 6 gm and different mixing ratios illustrated in Table 1 were produced using (CPS) and (BRH) in order to remove (BB-D) from the simulated wastewater samples.

Table 1: Mixing ratio of (CPS) and (BRH) hybrid adsorbents

BRH mixing ratio	Wight of adsorbents(gm)	
	(BRH)	(CPS)
100%	6	0
80%	4.8	1.2
60%	3.6	2.4
40%	2.4	3.6
20%	1.2	4.8
0 %	0	6

2.2 Characterization of Adsorbents

2.2.1 Elemental Analysis

Elemental analysis of carbon (C), hydrogen (H), nitrogen (N), oxygen (O), and sulphur (S), contained in (BRH) and (CPS) were determined by using Flash EA Elemental Analyzer (CHNS – O). In particular, an empty tin cup was placed in analytical balance which was normalized to zero. Then, the sample was added to the tin cup and weight was regulated until it is in the range of 2.5 – 3.0 mg. The weight of the sample was recorded on data sheet and in sample table. After that, edge of the tin cup was fold over to seal the sample. Then, the sealed sample was placed into tray in collect slot.

2.2.2 Surface Morphology Analysis

Field Emission Scanning Electron Microscope (FESEM) was also used to study the surface morphology of the (BHR) and (CPS) samples. Specifically, FESEM studies were carried out by using a ZEISS SUPRA 40VP FESEM at an electron acceleration voltage of1 kV and magnification of 1000 times. Prior to scanning, samples were coated with a thin layer of gold using a sputter coater to increase their electrical conductivity to release the electrons absorbed from the beam, reducing or avoiding the occurrence of charging.

2.2.3 BET Surface Area

Surface area analysis was undertaken using a Micromeritics Chemi Sorb Surface Area Analyzer. The Brunauer–Emmett–Teller method (BET) was applied to estimate the specific surface area (S_{BET}).Prior to analysis; each catalyst sample was enclosed in glass cell and evacuated or purged in the inert gas to ensure that there was no adsorbed moisture. Theoretically, the BET method is based on adsorption of nitrogen gas on a surface of BRH and CPS adsorbents. The amount of gas adsorbed at a given pressure allows surface area determination.

2.3 Preparation of Simulated Wastewater

0.2 g of (BB-D) was dissolved in 250 ml distilled water. The resulting dissolved water was then agitated under stirring at 600 rpm for 5 minutes. The simulated wastewater was prepared by diluted dye solution with distilled water until the volume of each sample reached 10 liter, so the initial concentration for each dye will be 20 ppm. The simulated wastewater samples were stored in chemical container.

2.4 Adsorption study

Six of adsorption studies were carried out by using hybrid adsorbent from Palm Shell activated carbon and Blended Rice Husk in single – layered fixed bed column. These experiments were considered as reference experiments in order to evaluate the effects of multi – layered fixed bed column and Blended rice husk, palm shell activated carbon treatments in terms of adsorption capacity. The reference experiments were conducted using a burette of 1.6 cm diameter. Figure 1 and 2 shows the schematic diagram of the experiments set-up used for adsorption studies. Six hybrid adsorbents with total amount of 6 grams and different mixing ratio as mentioned above were prepared and drying in the Oven at temperature of 30C^0 before being used in order remove the moisture. Then each hybrid adsorbent was loaded from the top of the burette and placed on 1cm thick of cotton at the bottom of the burette in order to hold the hybrid adsorbent. The burette was regularly shaken while being loaded with hybrid adsorbent to minimize void volume as well as air gaps and allow settling by gravity. The top surface of each hybrid adsorbent was covered with 1cm thick of cotton to prevent the spread out of the hybrid adsorbent when dye solution was filling to the burette. After that the burette was charged with simulated wastewater in the down flow mode manually. Then, the first set experiments were carried out by using six hybrid adsorbents from the Palm Shell activated carbon and Blended Rice Husk in the multi – layered fixed bed column. The first set experiments were conducted by following the similar experimental procedures with the single layered fixed bed column, except that the burette was packed with each hybrid adsorbent in three layers of bed, each having 2 grams of hybrid adsorbent. The distances between layers inside the burette were fixed to 2 mL.The treated wastewater samples were then collected at certain time intervals until the volume for each sample is reached 10 ml. Adsorption capacity was evaluated by means of area under the graph analysis and removal efficiency.

2.5 Data Analysis

2.5.1 Area under the Graph Analysis

Adsorption performance of all experiments was evaluated by area under the graph analysis. Therefore, the area under the graph was determined by the application of the trapezoidal rule of integration. Since the number of interval during 180 minutes was different for every adsorbent, the non-uniform trapezoidal rule (Equation 1) was adopted throughout this study.

$$\int_a^b f(x)dx \approx \frac{1}{2}\sum_{k=1}^{N}(x_{k+1}-x_k)(f(x_{k+1})+f(x_k)) \qquad (1)$$

Where, a: Value at first point of time

b: Value of last point of time.

N: Number of interval.

k: Point of value .

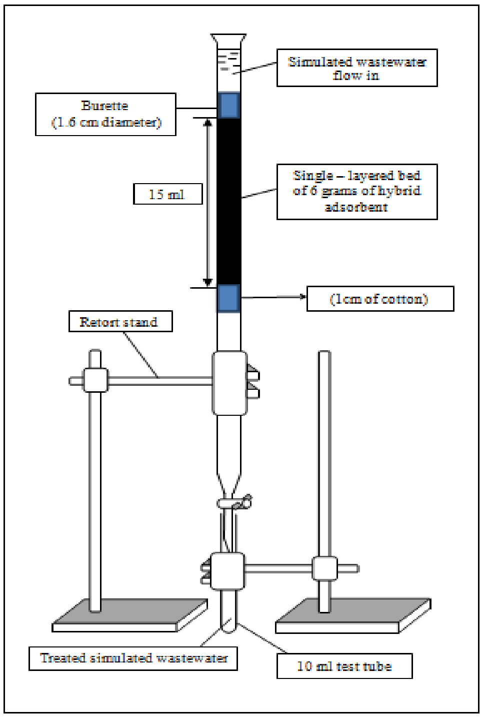

Figure1: Schematic diagram of experimental set up for single – layered fixed bed column used in adsorption study

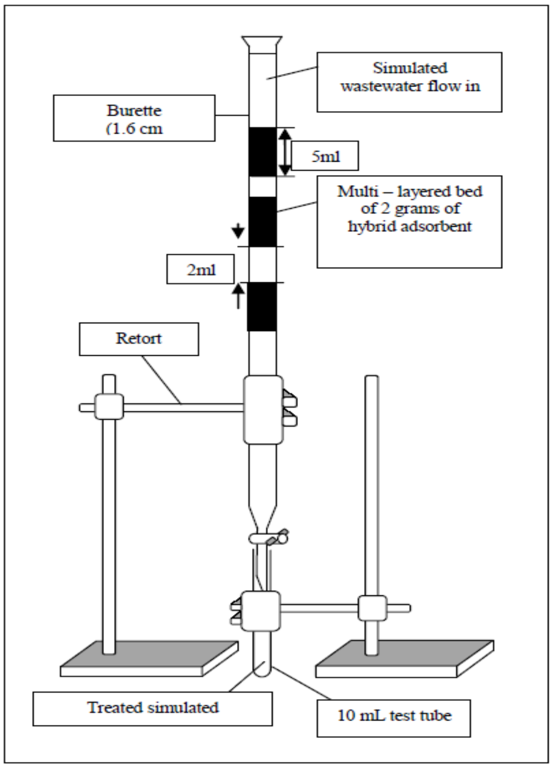

Figure 2: Schematic diagram of experimental set up for multi – layered fixed bed column used in adsorption study

2.5.2 Removal Efficiency Analysis

Adsorption behaviors of dye towards each type of hybrid adsorbents were studied by evaluating percentage removal efficiency from Equation (2).

$$\%\,\text{Removal} = \left[\frac{C_0 - C}{C_0}\right] \times 100 \qquad (2)$$

Where,

C_0 : Initial concentration of dye.

C: Concentration of dye after adsorption at any time.

3. Results and Discussion

3.1 Characterization of Adsorbents

3.1.1 Elemental Analysis

Regarding to BRH adsorbent, mean percentage values for carbon (C), hydrogen (H), oxygen (O), and nitrogen (N) were 36.96 4.58 , 55.14, and 1.14 respectively as shown in Table 2. The elemental composition of raw palm shell (RPS) and CPS adsorbents was shown in the same Table 2. The carbon percentage increases from 47.3% to 55.15% after the RPS was carbonized where it underwent conversion into a carbon-containing residue because of high temperature. Moreover, the moisture content and volatile compound were removed, clearing the path for adsorption and increase the adsorption efficiency.

Table 2: Elemental percentage of different types of adsorbents

	C	H	N	O	S
BRH (%)	36.96	4.58	1.14	55.14	0
RPS (%)	47.3	5.45	47.13	1.52	0
CPS (%)	55.1	1.12	39.6	2.82	0

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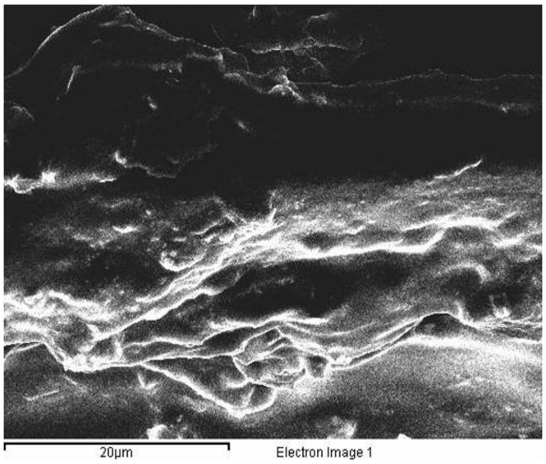
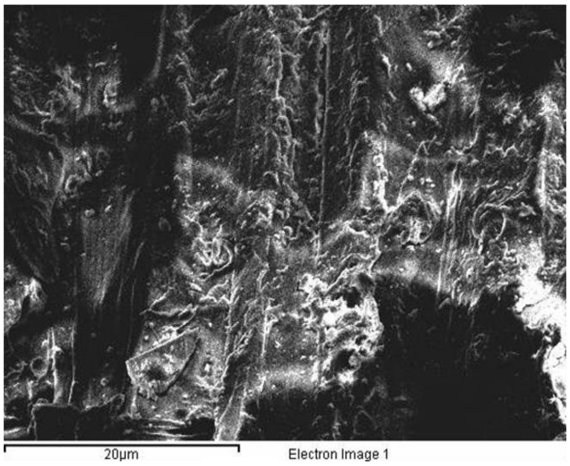
3.1.2 Surface Morphology

Figure 3 (a) and (b) presented the FESEM images of raw rice husk RRH and blended Rice Husk BRH respectively. Micrographs show considerable changes in morphology of rice husk adsorbent after expose to mechanical treatment. In particular, the surface of BRH was much rougher and highly heterogeneous than that of RRH, demonstrating the effect of size reduction. The heterogeneous surface of BRH provided more exposed surface area of rice husk adsorbent towards highly potential adsorption of dyes.



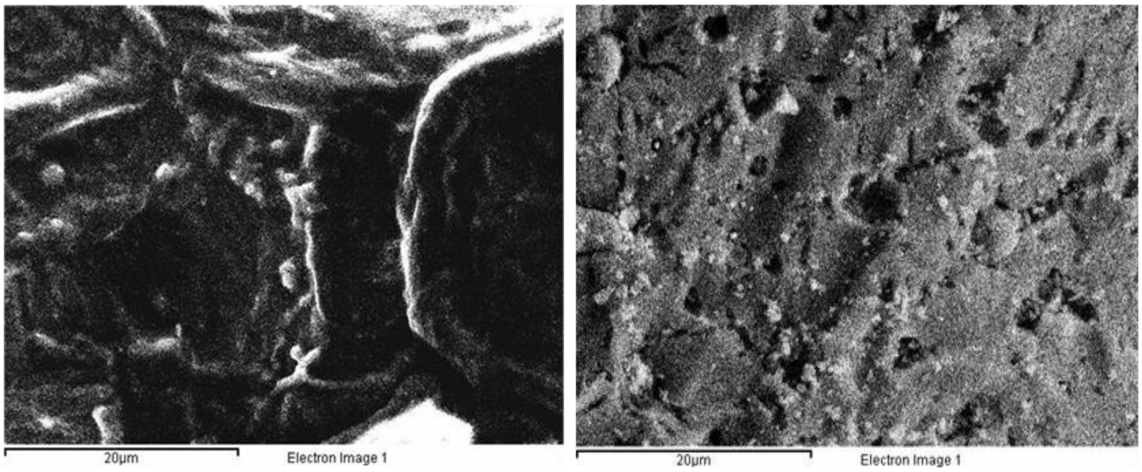
(a)



(b)

Figure 3: Surface Morphology of (a) Raw Rice Husk, (b) Blended Rice Husk adsorbent

The surface morphology of raw palm shell RPS and carbonized Palm Shell CPS is presented by Figure 4 (a) and (b) respectively. Figure 4 (a) presents the microphotograph of RPS under 3000x magnification. It was observed that the pore on the surface is hardly seen. This may be attributed to a thin layer of volatile compound and moisture exists on the top surface and covers the pores. However, the adsorption can still occur on the surface but in limited space. Meanwhile, in CPS adsorbent Figure 4 (b), pores are visible under 3000x magnifications. The thin layer appeared previously in Figure. 4(a) seems to disappear, due to the effect of carbonization. The carbonization of palm shell produces pores and eliminates volatile compound, moisture and other elements to generate clearer surface and larger surface area to provide an area for adsorption.



(a) (b)

Figure 4: Surface morphology of (a) Raw Palm Shell, (b) Carbonized Palm Shell adsorbent

3.1.3 BET Surface area record

A BET surface area analysis was performed on BRH and CPS adsorbents. Results are provided in Table 3.The specific surface area of BRH adsorbent was found to be 5.12m^2/g. This value was significantly greater than previously reported values for raw rice husk RRH (Aydin et al.2008; Christie, 2007; and Luo et al.2011). Whereas, the high surface area of CPS adsorbent (279.24 m^2/g) is mainly attributed to the combustion of palm shell organic components leaving several pores.

Table 3: BET surface area of adsorbents.

Adsorbents	BET surface area (m^2/g)
BRH	5.12
CPS	279.24

3.4. Evaluation of adsorption capacity

Area under the graphs analysis was performed by applying Equation (1) to evaluate the adsorption performance of aforesaid experiments. The concentration of (BB-D) in synthesis waste water for each mode of study was determined using six hybrid adsorbents with different mixing ratios of CPS and BRH and plotted accordingly. After plotting the graph, area under curve for each dye was determined by trapezoidal rule within contact time of 180 minutes. The lowest area under the graph represents the highest efficiency.

3.4.1 Adsorption of (BB-D) in single-layered fixed bed column.

The adsorption graph of (BB-D) using six hybrid adsorbents is illustrated in Figure 5. Since the lowest area under the graph represent the highest removal efficiency. It is clear that, (BB-D) is strongly adsorbed by 100% (BRH) and 0 % (CPS) hybrid adsorbent with the lowest area under graph and highest removal efficiency which are represented in Table 4 as 575.95, and 92.92% respectively. Meanwhile, all other hybrid adsorbent ratios exhibited low removal efficiency of less than 88 % through the contact time of 180 minutes. However, the area under the graph and removal efficiency for adsorption of (BB-D) for all hybrid mixing ratios is presented in table 4 below. As shown in Figure 5, desorption of the dye occurred with the 0% (BRH) - 100% (CPS) hybrid adsorbent at the 98 minute Probably due to the low interaction of dye with surface and longtime of adsorption, thus releasing the adsorbent into the wastewater it required the change of adsorbent for further adsorption process.

Table 4: Area under the graph and removal efficiency for adsorption of (BB-D) in single-layered fixed bed column

Hybrid mixing ratio	Area under graph	Removal efficiency%
100% (BRH) - 0% (CPS)	575.95	92.92
80% (BRH) - 20% (CPS)	1357.09	79.32
60% (BRH) - 40% (CPS)	3309.70	54.76
40% (BRH) - 60% (CPS)	5624.20	n/a
20% (BRH) - 80% (CPS)	1969.69	59.57
0% (BRH) - 100 % (CPS)	1196.25	87.53
Total area	14032.91	

Note: n/a refers to increment of Concentration of dye after adsorption.

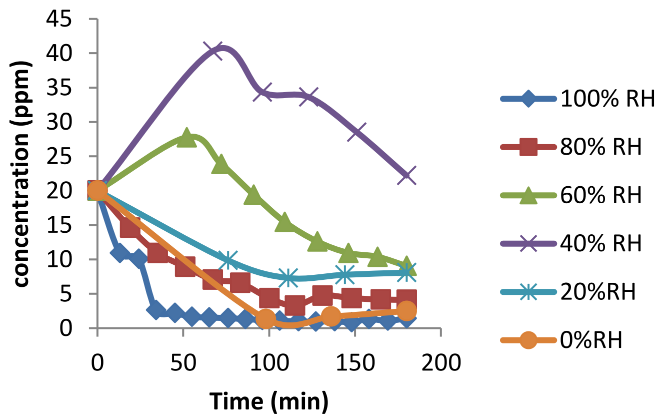


Figure 5: Adsorption of (BB-D) by six types of hybrid adsorbents in single-layered fixed bed column.

3.4.2 Adsorption of (BB-D) in multi-layered fixed bed column.

Particularly, according to the adsorption graph of Basic blue 90 by six hybrid adsorbents in multi-layered fixed bed column which is illustrated in Figure 6, it is shown that there was some improvement in removal efficiency of only two adsorbents which are100%RH and 100% CPS (0%RH) compared with single layered column adsorption .The removal efficiency never achieved 100% within contact time of 180 minutes by all hybrid adsorbents and represented by 40 % RH hybrid adsorbent < 60 % RH hybrid adsorbent < 20 % RH hybrid adsorbent < 80 % RH hybrid adsorbent < 0 % RH hybrid adsorbent < 100 % RH hybrid adsorbent with percentages of 6.25, 35.41, 55.26, 65.31, 89.76 and 97.17 respectively.

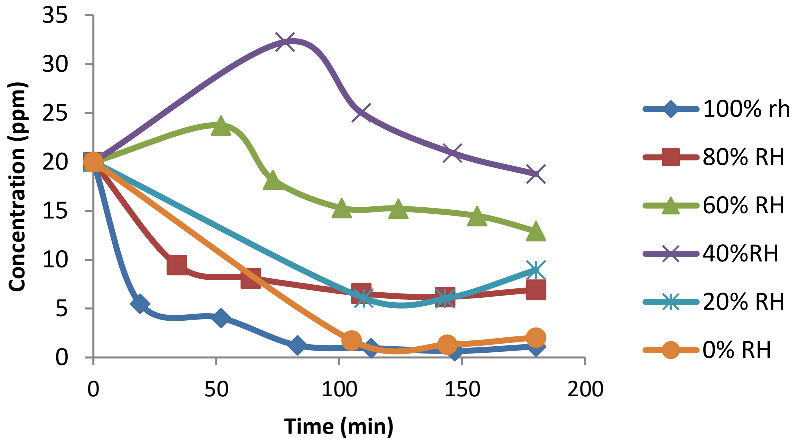


Figure 6: Adsorption of (BB-D) in multi-layered fixed bed column by six types of hybrid adsorbents.

Furthermore, table 5 shows the same trend for area under the graph reduction by all types of hybrid adsorbents.

Table 5: Area under the graph and removal efficiency for adsorption of (BB-D) in multi-layered fixed bed column

Hybrid mixing ratio	Area under the graph	Removal Efficiency%
100% (BRH) - 0% (CPS)	571.05	97.17
80% (BRH) - 20% (CPS)	1553.26	65.31
60% (BRH) - 40% (CPS)	3201.21	35.41
40% (BRH) - 60% (CPS)	4451.26	6.25
20% (BRH) - 80% (CPS)	1910.15	55.26
0% (BRH) - 100 % (CPS)	1262.40	89.76
Total area	12949.36	

From the results shown in tables (4, 5) it is clear that the removal efficiency of 100%RH and 100% CPS (0%RH) adsorbents in multi – layer column is significantly increased compared with single layer column adsorption. It can be conclude that, 100%BRH absorbent in multi-layered fixed bed column exhibited higher surface area compared to other mixing absorbents. The factors for high performance of adsorption are: void space between adsorbent, pore size, and particle size. The use of fixed bed column adsorption had enhanced the removal compared to many other studies by batch adsorption. The main idea to increase upper cross section area of pores, wall of pores and bottom area of pore to reach high performance by using all particle of modified adsorbent and reduce vacant spaces between particles and the advantages of using multi – layered over single layered column that is its design is able to overcome the limitation of single – layered fixed bed column practically regarding to vacant spaces. In single layered bed, the wastewater droplet tends to flow into less restrictive area, which is known vacant space. Vacant space in the bed can be defined as space in the bed which contains no occupied adsorbents. The presence of vacant space in the bed causes the distribution of wastewater flow inside the bed to be non – uniform. Besides that, the non – uniform distribution in the bed is also caused by high pressure. As a result, the contact time (residence time) between (BB-D) solutes and rice husk and carbonized palm shell hybrid adsorbent will be less which then reduced the probability of the solute molecules reaching an available adsorption site. By dividing the layer into three layers, the adsorption performance increased because each of wastewater droplets will be redistributed as it enters the new layer of fixed bed. The multi – layered arrangement also lead to reducing pressure inside the bed. Thus, the probability of solute molecules to reach another part of adsorption sites will be gradually increased.

4. Conclusion

Hybrid adsorbents were produced by mixing different ratios of BRH and CPS adsorbents and studied via single and multi-layered fixed bed column for dye removal. To evaluate the adsorption capacity of these hybrid adsorbents, (BB-D) was dissolved to make simulated wastewater. In single and multi-layered fixed bed column the results demonstrated that BRH adsorbent with no CPS expedited a highest removal efficacy of 92.92 and 97.17 respectively, with lowest area under graph of 575.95 and 571.05 respectively in 180 minute contact time. It can be conclude that, treatment of rice husk with carbonized palm shell did not reveal any beneficial effect. Blended rice husk was more effective compared to all mixed ratios used as adsorbent. For future studies, it is suggested to investigate the adsorption capacity of studied adsorbents for removal Basic Blue 90 dye from synthetic waste water by using physically and chemically modified adsorbents with different types of solvents in order to increase upper cross section area of pores, wall of pores and bottom area of pore to reach high performance by using all particle of modified rice husk and reduce vacant spaces between particles.

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