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## Synthesis of surfactant modified activated carbon (SMAC) from rice husks as Ni(II) and Cr(VI) adsorbent

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**Abstract.** Research on the synthesis of surfactant modified activated carbon (SMAC) from rice husk, investigation of the amount of surfactant added, contact time of surfactant on activated carbon, and the optimum condition of Ni(II) and Cr(VI) adsorbed by SMAC has been done. The carbon-making process was carried out by pyrolysis method where the carbon was activated using KOH for its effectivity in forming new pores and enlarging the surface area so that it can be used as an adsorbent. The activated carbons that had adsorbed sodium lauryl sulphate (SLS) surfactants would produce SMAC (Surfactant Modified Activated Carbon) with a more hydrophilic and negatively charged surface. SMAC can be applied as Ni(II) and Cr(VI) metal ion adsorbents. This research aims to determine the optimum KOH concentration for optimum activation process and optimum SLS concentration in the manufacture of SMAC, determining the adsorption efficiency of SLS surfactant by activated carbon in addition to determining the efficiency and the SMAC adsorption capacity of Ni(II) and Cr(VI). The results showed that the optimum concentration of KOH was 50%, the optimum concentration of SLS 60%, contact time was 4 hours and the adsorption efficiency was 99.69%. The adsorption efficiency of Ni(II) and Cr(VI) by SMAC were 95.96% and 99.49%, respectively.

**Keyword:** adsorption, SMAC

### 1. Introduction

Surfactant Modified Activated Carbon (SMAC) is a product that is produced from the process of modifying activated carbon with surfactants so as to increase the adsorption capacity of specific ions [1]. Modification of activated carbon using surfactants can improve the efficiency of activated carbon adsorption on metal waste adsorbates [2]. In principle, the SMAC system changes the surface of activated carbon from hydrophobic (non-polar) to hydrophilic (polar) properties [3]. In the manufacture of SMAC, there are several types of surfactants that can be utilized, which are anionic, cationic, and zwitterionic surfactants. The adsorption capacity of activated carbon which adsorbs anionic surfactant sodium lauryl sulfate (SLS) ( $0.89 \text{ mmol g}^{-1}$ ) was greater than that modified by cationic surfactant, cetyltrimethylammonium bromide (CTAB) ( $0.51 \text{ mmol g}^{-1}$ ) [4]. Modified SMAC with anionic surfactant can be applied to adsorb heavy metal cations in the waters. Ionic pollutants are generally adsorbed through electrostatic interactions or ion exchange on the surface [5].

One chemical solution that can be used as a carbon activator is KOH solution. The KOH activator has the advantage of being an activating agent [6] to produce activated carbon with size of pores that are predominantly microporous.

In the study of Gao *et al.* [7], activated carbon made from black water lignin activated using KOH activator was used to adsorb  $\text{Ni}^{2+}$  ions with an adsorption capacity of  $15,541 \text{ mg g}^{-1}$ . Hexavalent



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chromium receives a lot of attention, however, for its toxic nature and its ability to cause cancer in humans. In addition, Cr(VI) is more easily absorbed by the body than Cr(III)[8].

This research will investigate the synthesis of activated carbon made from rice husks activated at 400°C and 1-hour activation time, with variations in KOH concentrations of 40%, 50%, 60%, 70%, and 80%. The activated carbon was modified with SLS surfactant with contact time variations of 3, 4, 5, 6 and 7 hours and SLS concentrations of 40, 50, 60, 70, 80 ppm, forming SMAC to adsorb metal ions Ni(II) and Cr(VI). Carbon activated with KOH activating agent can eliminate volatile organic compounds such as alcohol, acids, aldehydes, etc. contained in the pores so that the carbon pores become open and the pore diameter and surface area of the adsorbent increase [9].

The purpose of this study is to determine the optimum concentration of KOH activator, as well as SLS concentration and optimum contact time. Moreover, determining the adsorption efficiency of the modified carbon process for the manufacture of SMAC in addition to the efficiency and adsorption capacity of metal ions Ni(II) and Cr(VI) by SMAC.

## 2. Experimental Methods

### 2.1. Equipment

A set of standard laboratory glassware, pyrolysis reactor, Mettler AT 200 scales, Isotemp 630F oven, eurotherm 2116 equipped furnace, UV-Vis spectrophotometer Shimadzu UV-1201, 100 mesh sieve, SAA Quatrachomenove 1200e, AAS Perkin Elmer.

### 2.2. Materials

Rice husks, sodium lauryl sulphate (SLS) surfactant, distilled water, methylene blue, concentrated H<sub>2</sub>SO<sub>4</sub>, NaOH solid, KOH solids, NaH<sub>2</sub>PO<sub>4</sub>·1H<sub>2</sub>O, NiSO<sub>4</sub>·6H<sub>2</sub>O, K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>, phenolphthalein indicator, chloroform p.a.

### 2.3. Preparation SMAC

Cleaned rice husks were carbonized through pyrolysis process at 300°C for 10 minutes. The carbonized product was activated using KOH concentrations of 40%, 50%, 60%, 70%, and 80% w/v for 1 hour at 420°C. The activated carbon obtained was washed with distilled water until the pH was constant. It was then dried, ground, and sieved with a 100 mesh sieve and underwent the SLS adsorption process. SLS adsorption aims to determine the optimum KOH concentration as an activator, where the SLS concentrations were varied to 40, 50, 60, 70, 80 ppm with time variations of 3, 4, 5, 6 and 7 hours so that the optimum concentration and contact time could be determined or the optimum SMAC was formed.

### 2.4. The application of SMAC

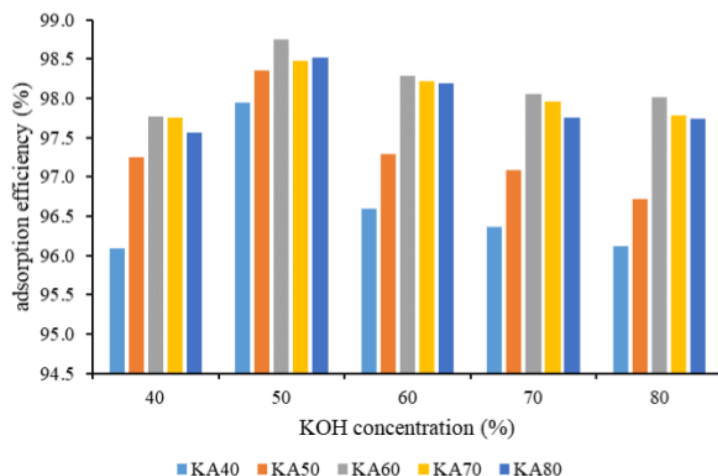
The resulting SMAC was contacted with Ni(II) and Cr(VI), at the initial concentrations of Ni(II) 40 ppm and the adsorption time of 300 minutes [10] while Cr(VI) 15% and the adsorption time of 30 minutes [11].

### 2.5. Characterization of SMAC

The SMAC which has been adsorbed SLS at optimum conditions (concentration and time of adsorption) characterized by a Surface analyser as a comparison is activated carbon with a 50% KOH concentration.

### 3. Results and Discussions

#### 3.1. Preparation of SMAC

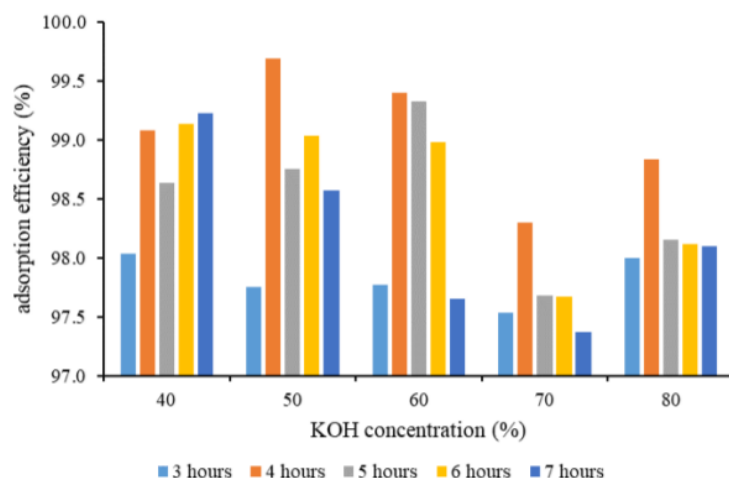


**Figure 1.** Correlation between adsorption efficiency, SLS concentration and concentration of KOH activator.

Figure 1 above shows the correlation between KOH concentration and surfactant adsorbed. The KA50 (activated carbon with 50% KOH) contacted with SLS Surfactant tended to have the highest peak of the graph at each of the SLS concentrations of 40, 50, 60, 70 and 80 ppm. In KA40, the concentration of SLS surfactant adsorbed by activated carbon was still considerably low. This is possibly because KOH with a concentration of 40% was unable to make more pores or there were still impurities in the carbon pores. At greater KOH concentrations (60, 70 and 80 ppm) carbon might severely erode and face damage causing the ability to adsorb SLS to decrease or for SLS to not be maximally adsorbed [12].

The initial concentration of surfactant adsorbed by the optimum activated carbon was 60 ppm with an efficiency of 98.75%. Above 60 ppm of SLS concentration, the efficiency experienced constant state or an insignificant decrease indicating that the activated carbon was saturated. This research determined the optimum conditions for adsorption which was only measured based on adsorption efficiency, so it was only determined based on the adsorbate.

Figure 2 shows the correlation of adsorption efficiency with KOH concentrations of 40%, 50%, 60%, 70% and 80%. The initial SLS surfactant concentration was 60 ppm and the SLS contact time variations were 3, 4, 5, 6 and 7 hours. The contact time between activated carbon and SLS is one of the factors that influence the adsorption process.



**Figure 2.** The correlation between adsorption efficiency, adsorption contact time and concentration of KOH activator.

From Figure 2, the optimum contact time of SLS surfactant is 4 hours at 50% KOH concentration with adsorption efficiency of 99.69%. At 3 hours of contact time carbon activated using KOH 40,50,60,70 and 80%, the adsorbed SLS was still small with an efficiency of 97-98%, this stated that not all activated carbon surfaces were covered by SLS. This shows that the adsorption ability of 50% activated carbon with 4 hours contact time is the best in adsorbing SLS surfactants, above 4 hours, the surface of the activated carbon was saturated by SLS or the adsorbed SLS might be re-desorbed.

Modification of activated carbon with SLS surfactants will produce SMAC. The addition of SLS surfactants to activated carbon can change the surface of activated carbon to be more hydrophilic and charged. This happens as the surfactants adsorbed onto the activated carbon through hydrophobic bonds. In other words, the surfactant hydrophobic groups will bond to the activated carbon and the hydrophilic groups will form the outer surface [13]. The charge of the activated carbon surface corresponds to the charge in the SLS (-) hydrophilic group. Thus, facilitating access for the active carbon to bind metal ions that are water soluble[14].

### 3.2. Adsorptions of Ni(II) and Cr(VI)

The resulting SMAC was exposed to Ni(II) solution with an initial concentration of 40 ppm for 300 min and Cr(VI) with an initial concentration of 15 ppm for 30 minutes. Based on Table 1, SMAC exposed to Ni(II) ion for 300 minutes had an adsorption efficiency of 95.97% while the adsorption efficiency of Cr(VI) for 30 minutes was quite high, which was 99.49%. This shows that the difference in charge will affect the adsorption efficiency. The greater the positive charge of a metal ion, the easier and higher the adsorbed metal ion.

**Table 1.** Efficiency of adsorption of Ni(II) and Cr(VI) ions by SMAC

Sample	Adsorption time(min)	Metal concentration	Adsorption Efficiency(%)
Ni(II)	300	40	95.97
Cr(VI)	30	15	99.49

there were differences in the initial concentration of Cr(VI), Ni(II) and adsorption times because this was the optimum concentration of Ni(II) 40 ppm and 300 minutes of adsorption time while Cr(VI) was 15% and 30 minute adsorption time [11].

**Table2.** Results of Active Carbon (KOH 50%) and SMAC Profile Analyses.

Profile	Size	CA (%)	SMAC (%)
Pore radius	15-20 Å	23,08	16,67
	21-90 Å	61,54	66,67
	>90 Å	15,39	16,67
Pore Volume	0,001-0,008 cc/g	-	25,00
	0,01-0,09 cc/g	30,77	75,00
	0,1-0,35 cc/g	69,23	-
Surface area	1-9 m <sup>2</sup> /g	-	25,00
	10-19,9 m <sup>2</sup> /g	-	58,34
	20-100 m <sup>2</sup> /g	30,77	16,67
	>100 m <sup>2</sup> /g	69,23	-

Activated carbon and SMAC profiles can be viewed from the pore volume and surface area. In Table 2, the pore volume is 0.1-0.35 cc/g and the surface area > 100 m<sup>2</sup>/g is not found at SMAC. This, in all likelihood, is because the surfactant has been adsorbed onto the pore and thus the pore volume decreased. Conversely, surfactants have occupied the space between layers of activated carbon. SMAC percentage for pore volume with 0.001-0.008 cc/g size increased, particularly the size 0.01-0.99 cc/g that increased up to 75% and the surface area of 10-19.9 m<sup>2</sup>/g also increased.

Despite the fact that SMAC has smaller surface area than activated carbon, it has a negatively charged active site on its surface so as to adsorb Ni(II) and Cr(VI). Based on the account above, activated carbon and SMAC are predominantly consisted of mesoporous pores because mesoporous radius ranges from 20-500 Å [15] while the radius of the metal ion ranges around 0.3-2 Å or more. This shows that adsorption occurs not only on active sites that are negatively charged but also occur within the pores.

Figure 3 implies that there are differences between pores of activated carbon and SMAC pores. The curve of the SMAC formed an open circle called the "ink bottle" pore which can take up adsorbate. These pores are wider on the inside than at the ends. Therefore, gas cannot enter until the pressure is raised to correspond to its capillary radius. After this pressure is raised the gas will condense so that the entire space is filled and produces a high pore volume. This situation leads to a hysteresis effect, which reduces pressure and cause the gas to leave the ends of the capillary with appropriate pressure, however the gas in the ink bottle section of the capillary will be trapped. This indicates that surfactants cause the formation of pores such as ink bottle. this is because SLS are adsorbed in the pore causing the pore to be closed and the pore surface getting narrower like a bottle neck.

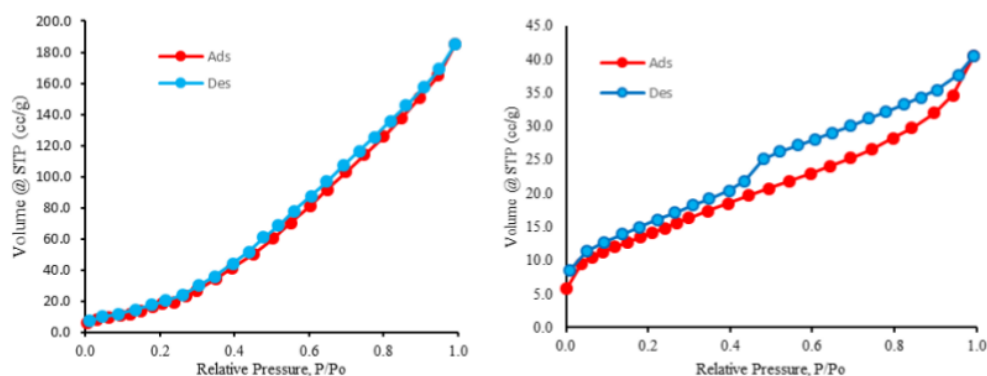


Figure 4. BET graph of (a) Active Carbon and (b) SMAC.

#### 4. Conclusion

The synthesis of SMAC from activated carbon made out of rice husk and SLS surfactant required 50% KOH and an optimum initial SLS concentration of 60 ppm with an optimum contact time of 4 hours resulting in efficiency ranging from 98.75 to 99.69%. The formed SMAC can adsorb Ni(II) by 95.75% with a contact time of 300 minutes and Cr(VI) at 99.49% with a contact time of 30 minutes. Cr(VI) and Ni(II) cations are adsorbed more in pores with a size of 0.01-0.09 cc/g and on the active side which is negatively charged from the hydrophilic head of SLS.

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