

## Irradiated Water-activated Waste Tyre Powder for Decolourization of Reactive Orange 16

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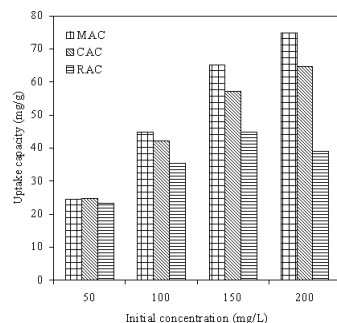
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### Graphical abstract



### Abstract

The present study was aimed to characterize the adsorptive properties of waste tyre powder based activated carbons for decolourization of reactive orange 16 (RO16). Waste tyre powder was activated through irradiated water environment (MAC). Comparison was made by conventional chemical activation using calcium chloride (CAC) and recovered calcium chloride from the first activation (RAC). Activated carbons were characterized according to surface area, morphology and functional groups. The values of surface area were recorded as 95.9, 111, 80.9m<sup>2</sup>/g for MAC, CAC and RAC, respectively. The decolourization of RO16 was observed to have the following order: MAC>CAC>RAC. Adsorption data for all activated carbons studied obeyed Langmuir isotherm for which the process could be described as monolayer adsorption. The kinetics data were well-fitted to pseudo-second-order model, suggesting the chemisorption process.

**Keywords:** Activated carbon; calcium chloride activation; irradiated water activation; reactive orange 16; waste tyre powder

### Abstrak

Kajian ini bertujuan untuk mencirikan sifat jerapan karbon teraktif dari sisa serbuk tayar sebagai penyahwarna reaktif jingga 16 (RO16). Sisa serbuk tayar telah diaktifkan melalui persekitaran air teriradiasi (MAC). Perbandingan telah dibuat dengan pengaktifan kimia biasa menggunakan kalsium klorida (CAC) dan kalsium klorida yang dikitar semula daripada pengaktifan pertama (RAC). Karbon teraktif telah dicirikan mengikut luas permukaan, morfologi dan kumpulan berfungsi. Jumlah luas permukaan dicatatkan sebagai 95.9, 111, 80.9m<sup>2</sup>/g masing-masingnya untuk MAC, CAC dan RAC. Penyingkiran warna RO16 didapati mempunyai tertib berikut: MAC>CAC>RAC. Data Penjerapan untuk semua karbon teraktif yang dikaji mematuhi isoterma Langmuir yang mana proses boleh digambarkan sebagai penjerapan selapis. Data kinetik memberikan penyuai terbaik bagi model pseudo-tertib-kedua, menunjukkan proses adalah penjerapan kimia.

**Kata kunci:** Karbon teraktif; pengaktifan air teriradiasi; pengaktifan kalsium klorida; reaktif jingga 16; serbuk tayar sisa

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### 1.0 INTRODUCTION

The release of dyes from textile industries into receiving water affects the aquatic creatures because some dyes are highly toxic and carcinogenic [1]. Dyes block the passage of sunlight into the stream, thus destroy the life cycle and food chain in water, and disrupt the biodiversity within. As water is a basic requirement in daily life, the deteriorate quality of water will bring bad consequences to human being.

Activated carbon adsorption is a preferred separation technique over other physico-chemical methods of dye removal because of its rich surface and pore properties [2-4]. However, precursors of activated carbon like coal and petroleum pitch are not renewable, while regeneration of the spent activated carbon is relatively expensive. This scenario has brought about searches for alternative carbonaceous precursors that are abundantly

available and low cost. One of the promising candidates under this category is waste tyre powder, a pyrolysis by-product of bulk waste tyre to obtain low-grade fuel.

Microwave heating is an emerging alternative to substitute conventional heating at high temperature in the preparation of activated carbon [5-6]. In microwave heating, energy is transferred into heat by the act of microwaves, i.e., heating from the interior of the material through dipole reorientation and conductive loss mechanisms, and so no contact between the material and the heating source is necessary [7-8]. Microwave heating in general offers rapid and efficient heat transfer, short treatment time, lower energy consumption and insignificant release of harmful gas [5-6].

The aim of this work was to investigate the potential of waste tyre powder-activated carbon prepared through microwave-induced water activation for decolourization of

reactive orange 16 (RO16). Chemical activation using calcium chloride and recovered calcium chloride were employed for comparison. The effects of initial dye concentration and contact time were examined and discussed.

## 2.0 EXPERIMENTAL

### 2.1 Materials

All chemicals were of analytical-reagent grade. Waste tyre powder was obtained from Bukit Batu Brickmills (M) Sdn. Bhd. Reactive orange 16 ( $C_{20}H_{17}N_3Na_2O_{11}S_3$ ; MW: 617.54;  $\lambda_{max}$ : 494) was used as model dye. Figure 1 shows the molecular structure of RO16. In aqueous solution, this anionic dye carries negative charges due to the presence of two sulphonate groups ( $SO_3^-$ ).

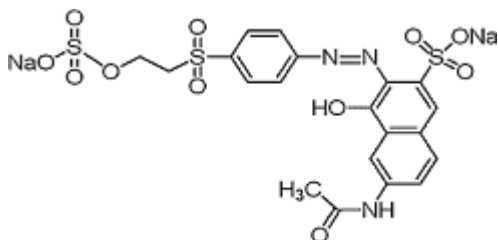


Figure 1 Molecular structure of reactive orange 16

### 2.2 Preparation of Activated Carbon

The waste tyre powder was first treated in furnace at 250°C for about 1.5 h to remove residual oil covering its surface that could prevent the solid to be well mixed with the activating agent.

**Irradiated water activation:** the precursor was soaked in distilled water, and activated under microwave at half magnitude of power intensity for 20 minutes. The resultant activated carbon was designated as MAC.

**Chemical activation:** the precursor was impregnated with calcium chloride at impregnation ratio (weight of activating agent/weight of precursor) of 1. Activation was done in furnace at 550°C for 1.5 h. The resultant product was washed using distilled water in a soxhlet unit to recover used calcium chloride for second activation. Similar procedures were repeated in second activation using the same weight of precursor as in the first activation. Activated carbons were designated as CAC and RAC for activation using fresh and recycled calcium chloride, respectively.

All activated carbons were dried in an oven prior to characterization and adsorption studies. Yield of activated carbon was determined from the weight of resultant product over the weight of treated waste carbon powder.

### 2.3 Characterization of Activated Carbon

The surface area of activated carbons was measured using surface area analyzer (Micromeritics PulseChemiSorb 2705, USA). The surface morphology was obtained using SEM instrument (Philips XL 40, Netherlands). Peaks at different wavelengths that correspond to surface functionalities were determined using FTIR instrument (Perkin Elmer Spectrum 2000 Explorer, USA).

## 2.4 Adsorption Studies

**Equilibrium isotherms:** Reactive orange 16 was employed as pollutant probe in adsorption. In batch adsorption, 0.1 g of activated carbon was added into conical flasks containing 50 mL varying concentrations of RO16 (5 to 300 ppm). The solution pH was not adjusted, and measured as  $5.8 \pm 0.3$  for all initial concentrations studied. The flasks were sealed, and the mixtures were allowed to equilibrate on orbital shaker at  $28 \pm 1^\circ\text{C}$  and 90 rpm for 72 hours. Thereafter, the solutions were filtered and the residual concentrations were determined using Visible Spectrophotometer (Biochrome Libra S6, UK) at a wavelength of 478nm. Equilibrium data were analyzed using commonly used isotherms, namely Langmuir [9-10] and Freundlich [11] models.

**Adsorption kinetics:** Fixed amount of 0.1g sample was added to different batches of 50 ml 200 mg/L of RO16 solution. The residual concentration was measured at different time intervals for 72 hours. Kinetics data were fitted to pseudo-first order [12] and pseudo second order [13] models, and the respective constants were determined.

## 3.0 RESULTS AND DISCUSSION

### 3.1 Characteristics of Activated Carbon

Table 1 shows the yield and surface area of activated carbons.

Table 1 Yield and surface area of activated carbons

Activated carbon	Yield (%)	Surface area ( $\text{m}^2/\text{g}$ )
MAC	72.3	95.9
CAC	82.3	111
RAC	77.8	80.9

In general, the activation of waste tyre powder gives a higher yield compared to other carbonaceous counterparts because the precursor is readily rich in carbon content [14]. The values reported in this work are somewhat greater compared to previous related works [15]. A decrease in yield indicates that there is burn-off of carbon material occurred in irradiated water activation. Results also show that the yield of MAC is slightly lower compared to that of CAC and RAC, because under microwave heating high temperature could be reached in seconds thus resulting in speeding up the release of tar or volatile matter compared to the conventional heating [16].

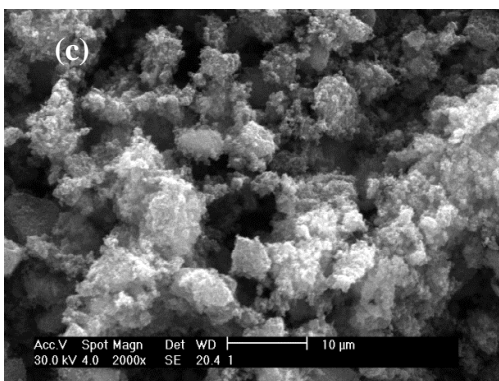
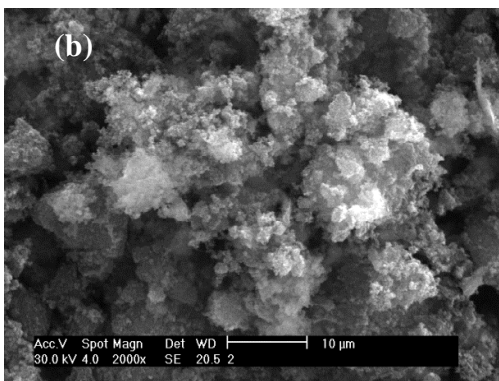
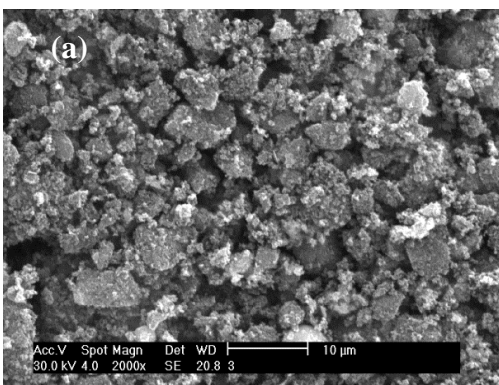
The values of surface area were recorded as 96, 111 and 81  $\text{m}^2/\text{g}$  for MAC, CAC and RAC, respectively. The surface area of MAC is as good as that of CAC, suggesting a potential of using irradiated water in activated carbon preparation [17].

It is generally understood that water and carbonaceous material are dielectric materials that can easily absorb the microwave radiation and have the ability to dissipate the energy (heat) [6-7]. At the microwave frequency of 2450 MHz and energy of  $1.02 \times 10^{-5} \text{eV}$ , the water molecules flip around 4.9 billion times per second [5-6]. A phase difference between the electric field and dipoles (water molecules) orientation causes energy to be lost in random collisions, and gives rise to dielectric heating [18-20]. The electro-magnetic field of microwave also induces an oscillating current to which the freely-move delocalized  $\pi$ -electrons of carbon material could couple with high-frequency electric field to produce hot spots known as microplasmas [6, 21]. The impacts of these combined heating thus making the development of pores possible at a shorter

period. Hence, it could be a promising method over conventional steam activation in activated carbon preparation [22].

From Table 1, the surface area of RAC is smaller than that of CAC probably due to used-up and evaporated calcium chloride in the first activation. Notwithstanding that, the utilization of recovered calcium chloride was still feasible to produce comparatively high surface area as in the first activation.

Figure 2 shows the SEM images of activated carbons. The formation of melt, vesicles, precipitates of inorganic salts and surface etching were observed on the surface of activated carbons. Under the same magnification, MAC demonstrated smaller particle size than CAC and RAC, probably due to the absence of chemical used in the activation. On the other hand, calcium chloride caused the particles to agglomerate. All activated carbons displayed irregular structures with micro-holes, cracks and crevices which confirmed the amorphous and heterogeneous structures [23].



**Figure 2** SEM images for activated carbons (a) MAC, (b) CAC and (c) RAC

The FTIR spectra of activated carbons are tabulated in Table 2. The peaks at different wavelengths qualitatively represent chemical structure and available functional groups on the surface of activated carbon. Obviously, all activated carbons possess similar spectra which indicate identical surface properties within their structure. The region between 3700-3200  $\text{cm}^{-1}$  shows the presence of  $-\text{OH}$  stretching in bonded and non-bonded hydroxyl groups, and water molecules [23]. The existence of hydroxyl groups,  $\text{C}=\text{C}$  and  $\text{C}-\text{O}$  bonds suggests the normal structure for activated carbon [24]. The region 1600-1500  $\text{cm}^{-1}$  corresponds to aromatic ring stretch of  $\text{C}=\text{C}$  in olefinic structure that also signifies the graphitic structures of activated carbon [25]. Prominent peaks that correspond to acidic surface functional groups such as carboxylic (1700  $\text{cm}^{-1}$ ) and lactonic (1740  $\text{cm}^{-1}$ ), however are absence from the spectra. In turn, this could promote the removal of RO16 as their presence would likely to inhibit the adsorption due to repulsive force from opposite ionic charge [14].

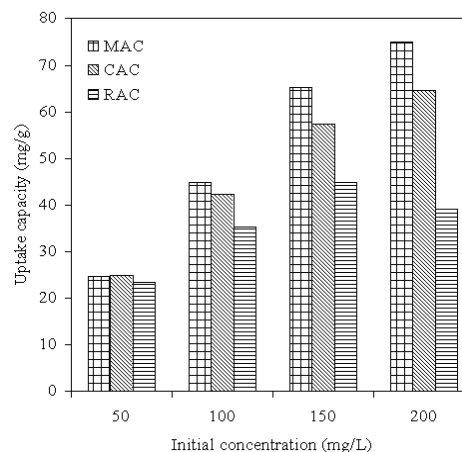
**Table 2** FTIR spectra of activated carbons

Wavelength ( $\text{cm}^{-1}$ )			Assignment
MAC	CAC	RAC	
3350-3700	3250-3750	3200-3750	O-H stretch
2800	2930	2850	C-H stretch (alkanes)
1500	1600	1505	Aromatic ring stretch
1100	1195	1095	C-O stretch (alcohol)

### 3.2 Equilibrium Isotherms

In adsorption, the values of equilibrium pH were found to be consistent for all activated carbons studied and were measured as  $6.4 \pm 0.2$ . The increased of solution pH indicates that the activated carbons are protonated and becomes positively charged [14]. The consistent values of equilibrium pH also suggest that the amount of protons adsorbed for each activated carbon is uniform although their surface area is different; ca. 0.0049 mmol/g proton was adsorbed. Therefore, it is adequate to utilize single component isotherm models to represent the adsorption data. In general, the increase of equilibrium pH occurs when the surface of activated carbon is absence from acidic functional groups [22]. It commonly results in a higher uptake of RO16 due to attractive force from the surface.

Figure 3 displays the removal of RO16 by activated carbons at different initial concentrations.



**Figure 3** Uptake of RO16 at different initial concentrations

The removal of RO16 by all activated carbons was found to increase with increasing initial concentration. At initial concentration of 50 ppm, activated carbons demonstrated an identical removal of RO16 at 24 mg/g. This is equivalent to more than 94% of RO16 removal. As initial concentration increases to 200 ppm, the decolourization of RO16 was found to follow the order: MAC>CAC>RAC.

The uptake of dye from aqueous solution is commonly driven by surface area and surface functional groups of activated carbon [25]. A higher surface area of CAC could be the reason for a greater RO16 removal compared to RAC. However, a superior uptake of MAC than that of CAC could be attributed to the changes in its surface activity as a result of irradiated water activation [26].

Two commonly used isotherm models for single solute adsorption, namely Langmuir and Freundlich were used to describe the adsorption data. The Langmuir isotherm [9-10] indicates monolayer adsorption onto a completely homogeneous surface, and is given as,

$$q_e = \frac{Q_0 b C_e}{1 + b C_e} \tag{1}$$

where  $Q_0$  (mg/g) is the maximum uptake per unit mass of adsorbent to form a complete monolayer on the surface of adsorbent, and  $b$  (L/mg) is a constant related to the affinity of the binding sites. The empirical Freundlich isotherm [11] based on sorption on a heterogeneous surface is given by,

$$q_e = K_F C_e^{\frac{1}{n}} \tag{2}$$

where  $K_F$  and  $1/n$ , are the Freundlich constants, i.e., maximum adsorption capacity and intensity, respectively. The  $1/n$  value ranging from 0 to 1 is considered to represent surface heterogeneity. It is also suggested that the  $n$  values ranging between 2 and 10 represent favourable adsorption process. The applicability of these models was deduced based on coefficient of determination ( $R^2$ ). Figure 4 shows the equilibrium adsorption of RO16 onto activated carbons, while the respective constants are tabulated in Table 3.

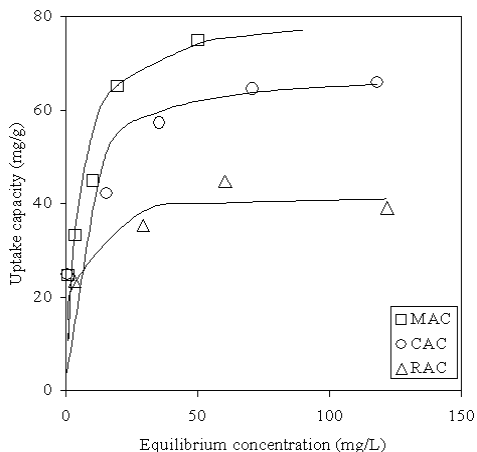


Figure 4 Equilibrium uptake of reactive orange onto activated carbons. Lines were predicted from Langmuir model

Table 3 Constants of Langmuir and Freundlich models

Activated carbon	Langmuir model			Freundlich model		
	$Q_0$ (mg/g)	$b$ (L/mg)	$R^2$	$K_F$	$1/n$	$R^2$
MAC	81.3	0.208	0.986	25.4	0.279	0.966
CAC	68.5	0.191	0.995	29.6	0.171	0.972
RAC	42.0	0.364	0.991	19.7	0.167	0.857

Both models were found to correlate the adsorption data well. The Freundlich model suggests that the uptake of RO16 by activated carbons is a normal adsorption onto heterogeneous surface. However, the values of  $R^2$  indicate that the Langmuir model is more fitted to a linear approximation. Therefore, the uptake of RO16 onto activated carbons could be adequately described as monolayer adsorption [14, 23]. The values of maximum adsorption capacity given in Table 3 are in agreement with the experimental data (Figure 4). Despite its lower maximum adsorption, RAC demonstrated a greater adsorption affinity,  $b$  compared to the other two activated carbons. Affinity of adsorption is commonly referred as a favourable removal at lower concentration. For example, a comparable uptake of RAC with its other two counterparts was observed at initial concentration of 50ppm (Figure 3). It implies the feasibility of using recovered calcium chloride for subsequent activated carbon preparation which suits the adsorption at lower dye concentration.

The adsorption data was further analyzed by separation factor,  $R_L = 1/(1+b \cdot C_0)$ , a feature of Langmuir model. The  $R_L$  value depicts the nature of adsorption process irrespective the shape of isotherm [27]. The adsorption is unfavourable if  $R_L > 1$ ; linear if  $R_L = 1$ ; favourable if  $0 < R_L < 1$ ; or irreversible if  $R_L = 0$ . Figure 5 illustrates the  $R_L$  profiles of activated carbons.

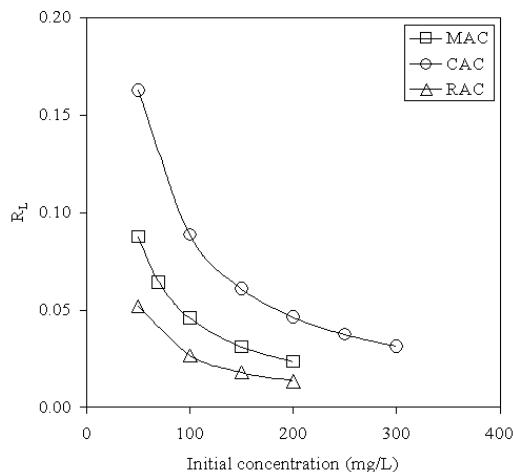


Figure 5  $R_L$  profiles of activated carbons

From Figure 5, the activated carbons displayed the  $R_L$  values ranging from 0.014 to 0.163, which fall under the favourable region of adsorption at all initial concentrations studied.

### 3.3 Adsorption Kinetic

A better understanding on the mechanism and rate of adsorption is important to design the adsorption process. For that purpose, high concentration at which the activated carbons nearly reach their saturation point was selected for kinetics study. This was done to imitate heavy discharge of effluent in the industry.

The kinetics data was evaluated using two kinetics models, namely pseudo-first order and Ho's pseudo-second order models. Pseudo-first order model [12] is given by,

$$q_t = q_e(1 - e^{-k_1 t}) \quad (3)$$

where  $q_t$  (mg/g) is the amounts of RO16 adsorbed at time  $t$  (h), and  $k_1$  ( $\text{h}^{-1}$ ) is the rate constant of first order adsorption. Ho's pseudo-second-order equation [13] based on chemical related adsorption is expressed as,

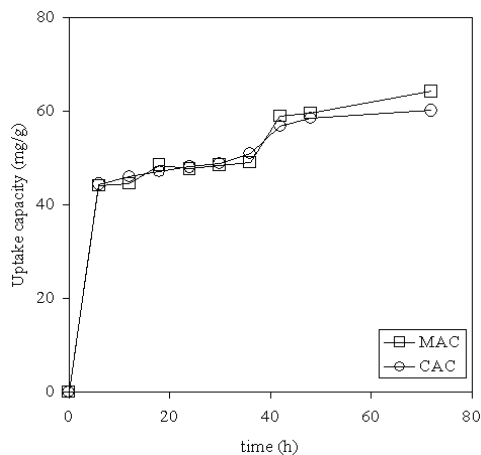
$$q_t = \frac{k_2 q_e^2 t}{1 + k_2 q_e t} \quad (4)$$

where  $k_2$  (g/mg.h) is the rate constant of pseudo-second-order adsorption. The initial adsorption rate,  $h$ , of pseudo-second-order as  $t$  approaching zero is defined as,

$$h = k_2 q_e^2 \quad (5)$$

The kinetics constants were solved using non-linear regression for sum of squared error (SSE) and coefficient of determination ( $R^2$ ).

Figure 6 shows the kinetics profiles of MAC and CAC, and their respective constants are tabulated in Table 4. From Figure 6, the uptake of RO16 was found to increase with increasing time. Both activated carbons showed a comparable trend of RO16 removal, and the equilibrium uptake over the studied period was reasonably tallied with that of adsorption isotherms (Figure 3). Rapid increase in adsorption by both activated carbons was observed at the first 6 hours, after which the uptake started to increase gradually. About half of RO16 concentration was able to be removed at the first 6 hours of adsorption. It is interesting to note a sudden steep gradient at a period between 36 and 42 hours. Similar profile was also reported by Sumari and co-workers [28] for RO16 concentrations of 40 and 100 ppm. This could be due to the interactions of adsorbent-adsorbate, and adsorbate-adsorbate as the adsorption progresses at higher concentration.



**Figure 6** Kinetics profiles of reactive orange 16 onto activated carbons

The SSE and  $R^2$  values in Table 4 explain that Ho's pseudo-second-order model is sufficient to describe the kinetics behaviour of RO16 onto MAC and CAC. Moreover, the predicted

$q_{e,cal}$  values from this model are in agreement with the experimental values. From Table 4, CAC demonstrated a slightly greater kinetics constant ( $k_2$ ) and initial adsorption rate ( $h$ ) compared to MAC. It is suggested that a higher surface area of the former could instigate more interaction probabilities for fairly quick removal of RO16. From the view point of kinetics, it can be postulated that the rate limiting step was chemisorption involving valency forces through sharing or exchange of electron between activated carbon and RO16 [29]. In addition, the affinity for adsorption as reported in Table 3 is expected to be driven in part by chemisorption of RO16 onto activated carbons.

**Table 4** Constants of pseudo-first order and pseudo-second order kinetics models

	MAC	CAC
$q_{e,exp}$ (mg/g)	64.3	60.2
Pseudo-first order model		
$k_1$ ( $\text{h}^{-1}$ )	0.226	0.260
$q_{e,cal}$ (mg/g)	53.7	52.8
SSE	321	197
$R^2$	0.887	0.925
Pseudo-second order model		
$k_2$ (g/mg.h)	0.00516	0.00685
$h$ (g/mg.h)	18.3	22.6
$q_{e,cal}$ (mg/g)	59.6	57.5
SSE	200	112
$R^2$	0.929	0.957

Table 5 summarizes some studies on the decolourization of RO16 by various adsorbents.

**Table 5** Decolourization of RO16 by adsorbents

Adsorbent	Maximum uptake (mg/g)	pH studied	Reference
Corynebacterium glutamicum	187	1	[30]
Quartenised sugar cane bagasse	22.7	7	[31]
EDTA modified rice husk	7.68	2	[32]
Activated carbon	8.75	Not reported	[33]
Copper Oxide Supported-Activated Carbon	15.8	Not reported	[33]
MgAlNO <sub>3</sub> layered double hydroxides	111	6.9	[28]
Fly ash based zeolite	0.58	Not reported	[34]
Ananas comosus leaves based activated carbon	112	8	[35]
Irradiated water-activated carbon powder (MAC)	81.3	6.4±0.2	Present study
CaCl <sub>2</sub> -activated carbon powder (CAC)	68.5	6.4±0.2	Present study

Won and co-workers [30] reported a considerably higher uptake of RO16 in acidic environment due to excessive protonation of adsorbent that would attract more negatively charged RO16 onto the adsorbent surface. However, the use of chemicals/acids in adsorption is likely to increase utility costs and also prompt unnecessary post-treatment. Compared to other

adsorbents, MAC and CAC demonstrated a better performance for RO16 decolourization at almost neutral pH; which will be a merit for industrial applications [4].

#### 4.0 CONCLUSION

Waste tyre powder-activated carbons were prepared through irradiated-water environment and calcium chloride activation. Irradiated water-activated carbon powder (MAC) showed a greater removal of reactive orange 16 (RO16) than calcium chloride-activated carbon powder (CAC) despite a slightly lower surface area of the former. This study demonstrated the feasibility of using water as activating agent under microwave irradiation. Activation of waste carbon powder using recovered calcium chloride (RAC) was still feasible for high intensity adsorption at lower RO16 concentration. Decolourization of RO16 onto activated carbons could be described by monolayer adsorption that is driven by chemical adsorption. Activated carbons prepared in this work could become a potential candidate for RO16 decolourization in industrial wastewater.

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