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Orange peel activated carbon produced from waste orange peels for adsorption of methyl red

Sneha Deshmukh [a], Niraj S. Topare [b,c,*], Sunita Raut-Jadhav [c], Prashant V. Thorat [a], Shantini A. Bokil [d] and Anish Khan [e,f]

[a] Department of Chemical Engineering, College of Engineering and Technology, Akola 444104, India
[b] School of Chemical Engineering, Dr. Vishwanath Karad MIT World Peace University, Pune 411038, India
[c] Department of Chemical Engineering, Bharati Vidyapeeth (Deemed to be University) College of Engineering, Pune 411043, India
[d] School of Civil Engineering, Dr. Vishwanath Karad MIT World Peace University, Pune 411038, India
[e] Chemistry Department, Faculty of Science, King Abdulaziz University, Jeddah 21589, Saudi Arabia
[f] Center of Excellence for Advanced Materials Research, King Abdulaziz University, Jeddah 21589, Saudi Arabia
*Corresponding author. E-mail: niraj.topare@mitwpu.edu.in; nirajtopare06@gmail.com

SD, 0000-0002-7015-2769; NST, 0000-0002-8875-5808; SR-J, 0000-0001-9984-0314; PVT, 0000-0001-8093-7351; SAB, 0000-0002-3278-2989; AK, 0000-0002-3806-5956

ABSTRACT

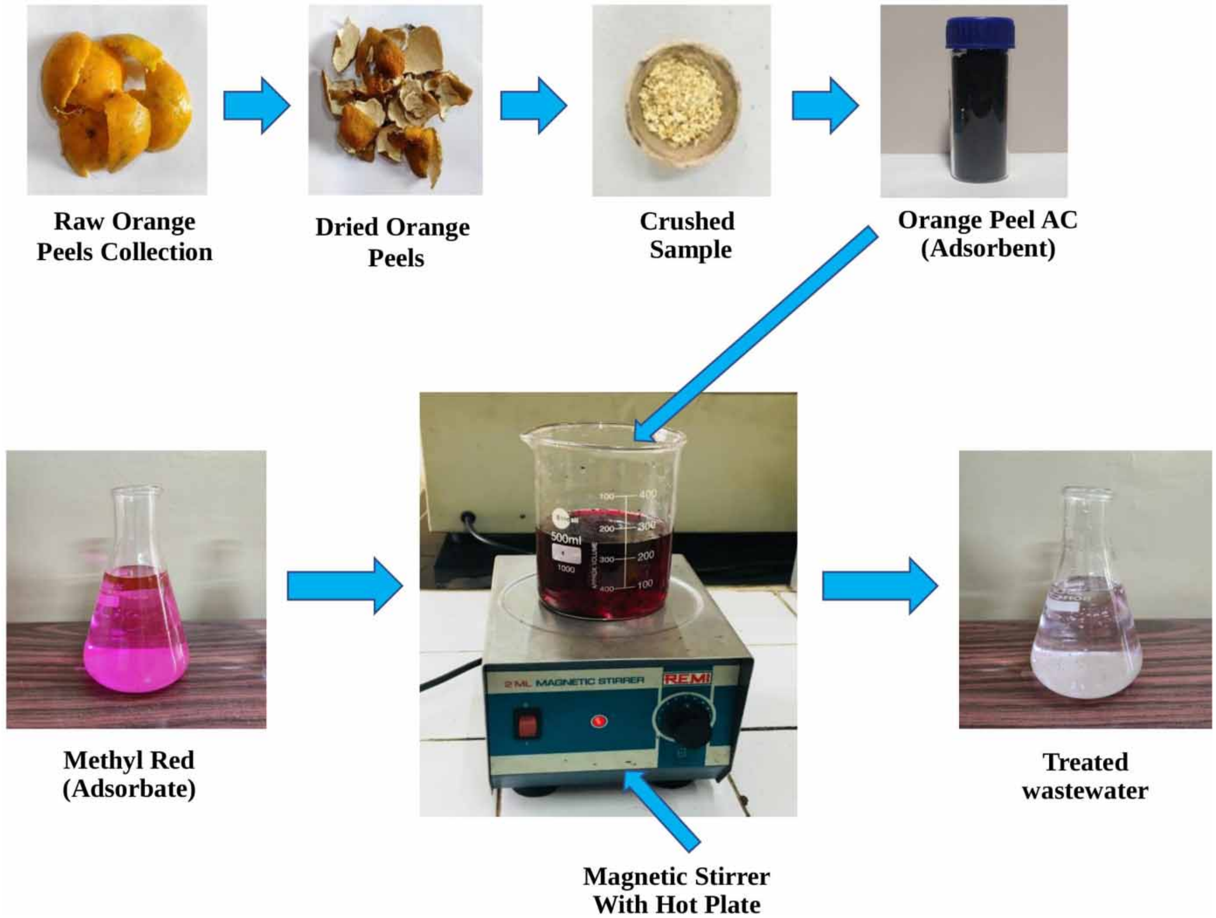
The existence of pollutants in the water is a very significant environmental problem that needs to be addressed. This work describes the development and testing of activated carbon made from orange peels, which is effective at removing methyl red (MR) from aqueous solutions, and thus provides a solution to this problem. Adsorbents made in the lab can be characterized by their bulk density, particle size, surface area, and proximate analysis. The surface area of the prepared adsorbent was 512.2 m^2g^{-1}. Standard procedures such as XRD, SEM, and FTIR analysis are also used to characterize prepared orange peel-activated carbon. Adsorbent dosage (0.25 to 1.25 g/L), MR concentration (100 to 400 mg/L), temperature (40 to 60 °C), contact time (10 to 60 minutes), and pH (3 to 11) were all examined in this experiment. At an amount of adsorbent of 1 g/L adsorbent, MR concentration of 100 mg/L, and a pH of 11, maximum adsorption has been observed. In order to analyze the results, adsorption models such as the Langmuir and Freundlich were applied. At 60 °C, the adsorption isotherm was found to fit the Langmuir model with 111.11 mg/g. The linear regression correlation coefficient, the R^2 value is 0.999. Analytical results showed that MR could be effectively removed by using AC made from waste orange peels as an adsorbent.

Key words: activated carbon, adsorption, isotherms, orange peels, process parameters

HIGHLIGHTS

- Orange peel activated carbon (AC) prepared from waste orange peels by carbonization and chemical activation method.
- Potential use of prepared orange peels AC for MR dye removal.
- Absorption process of orange peels AC fits Langmuir and Freundlich isotherms.
- Adsorption of MR on orange peels AC and commercial AC was compared.
- Adsorption of MR dye using orange peels AC is an efficient approach.

GRAPHICAL ABSTRACT



INTRODUCTION

Adsorption is a popular and efficient physical method of separation that may be used to get rid of or reduce the concentration of a wide variety of dissolved contaminants (organics and inorganics) in an effluent (Ali *et al.* 2012). It has recently come to light that activated carbon, also known as AC, is a well-known adsorbent that can be utilized effectively for the removal of a wide variety of contaminants from air, soil, and liquids. This development is very noteworthy. The majority of adsorption takes place on the pore walls of particles, which is why sorbents are typically classified as porous solids (Barna *et al.* 2013). AC, silica gel, activated alumina (which can absorb moisture), zeolites and molecular sieves, and synthetic resins are some examples of materials that can operate as adsorbents. AC is particularly effective at removing organic compounds. When it comes to removing organic, inorganic, and biological pollutants, AC is the most effective adsorbent that is of importance in the treatment of water and wastewater (Bharathi & Ramesh 2013). In recent years, it has been used more frequently for the purpose of preventing environmental pollution, and anti-pollution legislation has led to an increase in sales of air conditioners that are designed to reduce pollutants in the air and water (Cardoso *et al.* 2011).

AC is a processed type of microcrystalline, non-graphitic carbon having an internal porosity. Because of its high porosity, large surface area, and strong surface reactivity, AC is a very versatile material (Bokil *et al.* 2020). AC's high specific surface area ranging from 450 to 2,000 m^2g^{-1} facilitates the physical adsorption of gases, vapors, and dissolved or dispersed compounds from liquids (Hameed *et al.* 2007). It possesses a huge number of very tiny holes, which gives it a vast inner surface and excellent adsorption characteristics. In water and wastewater treatment, they effectively absorb organic, inorganic, microbiological, and biological pollutants (Akar *et al.* 2009; Balci *et al.* 2011).

The synthesis of ACs from biomass is an attractive technique to get carbonaceous compounds from food industry waste (Bokil & Rai 2016). Carbon materials are employed widely in numerous processes (Kadirvelu *et al.* 2001). The cost of using biowaste is rising. Using difficult-to-dispose-of waste biomass can benefit the environment. ACs from biomass are efficient and cost-effective compared with commercial carbons. Natural resources are widely employed as carbonaceous precursors (Topare & Bokil 2021). Orange peels are one of the waste materials that are utilized as precursors in the production of ACs. The present study involved the evaluation of different conditions (adsorbent dosage (0.25–1.25 g/L), MR concentration (100–400 mg/L), temperature (40–60 °C), contact time (10–60 min), and pH (3–11) in the MR adsorption efficiency). The data demonstrate that the orange peel-derived material possesses excellent adsorption capabilities, making it a promising option for actual use in wastewater treatment.

MATERIALS AND METHODS

Preparation of AC

The unprocessed components (orange peels), which are necessary for the production of AC, will be procured from local juice shops. After collecting orange peels, they were first washed to remove any dirt, then dried, then crushed using an electrical grinder, and then sieved using a mesh size of 60. The production of AC requires the use of a chemical activation agent, and several acids have been investigated and reported as potential agents. The glassware was cleaned with distilled water, and all of the chemicals that were utilized were of an analytical (AR) quality. The materials were carbonized at a temperature of 250 °C for 2 h before being allowed to cool at ambient temperature. After collecting and cleaning the samples, 30 g of each were combined with 30 ml of zinc chloride ($ZnCl_2$) at a 1:1 ratio and kept in a SS container. After heating in a muffle furnace at a temperature of 500 °C for 1 h, the container made of SS was removed. The AC samples were washed to remove any remaining residue from the preparation process. The procedure of washing was continued until a pH level of 7 was reached. After that, the samples were placed in an oven set at 100 °C in order to remove any trace of moisture.

Experimental procedure

The purpose of the experiments was to investigate the adsorption of MR using orange peel AC as the adsorbent. Studies of adsorption in batches are carried out by monitoring a variety of essential parameters such as adsorbent dosage (0.25–1.25 g/L), MR concentration (100–400 mg/L), temperature (40–60 °C), contact time (10–60 min), and pH (3–11). A batch reactor was where all of the adsorption studies were carried out. Before and after adsorption, MR dye concentrations in wastewater supernatant were measured with a UV–Visible spectrophotometer (Amin 2009). After that, the concentration in the effluent was calculated with the help of a standard calibration curve. Each experiment was carried out multiple times under controlled settings, and the results of the averages were used in subsequent calculations. Figure 1 represents the experimental setup for the batch adsorption study.

RESULTS AND DISCUSSION

Characterization of orange peels AC

AC made from orange peels was the adsorbent that was utilized in this experiment. The physicochemical properties (typical properties, proximate and ultimate analysis) of orange peel AC are utilized for the purpose of determining its characterization (Armagan *et al.* 2003; Topare *et al.* 2020). SEM is used to examine the surface morphology of orange peel AC samples. Using Cu$K\alpha$ as the radiation source, XRD was performed using an X-ray diffractometer. The functional groups were identified by recording FTIR spectra in the region of 4,000–500 cm^{-1}.

Brunauer–Emmett–Teller (BET) analysis was performed using Quantachrome Instruments v3.01 (Autosorb iQ Station 1). The BET surface area, pore volume, and pore diameter of the orange peel AC were measured by nitrogen adsorption methods. A thermogravimetric method, or 'loss on drying', is typically used to determine the moisture content. In this method, the sample is heated, and the weight loss from moisture evaporation is measured. The combination of a drying oven and a balance, along with a moisture analyzer, is a common moisture analysis technology. A small quantity of AC was weighed and then transferred to a glass dish. After that, it was heated for 2 h in an oven at a temperature of more than 100 °C. The glass dish was taken out of the oven and allowed to cool. The weight of the dried sample was determined after it had cooled. According to Ashtaputrey & Ashtaputrey (2016), the equation was used to calculate the moisture content (wt.%).

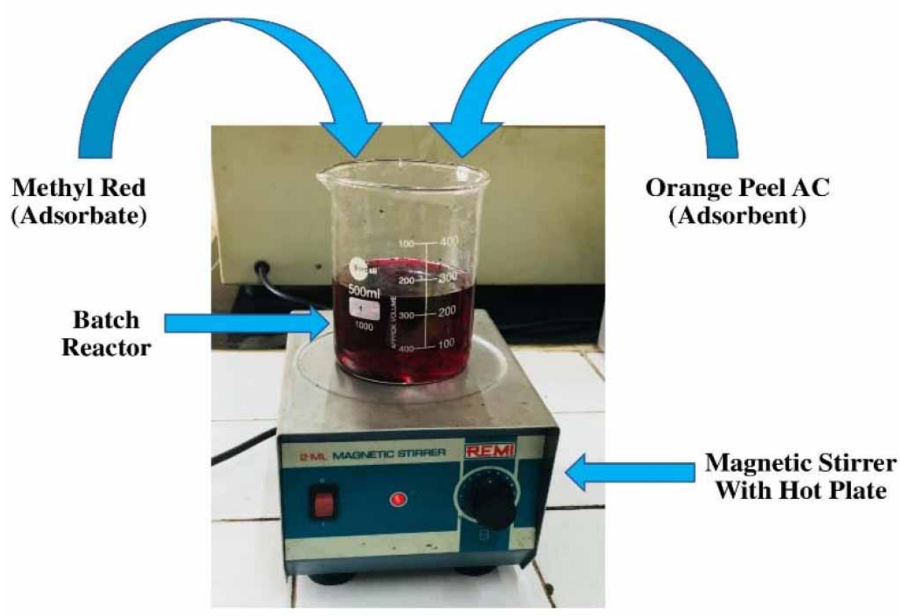


Figure 1 | Experimental setup for batch adsorption study.

The volatile matter content was measured directly in the automated proximate analyzer. When a sample of furnace oil is burned to completion, the incombustible residue is reflected by the ash content. High-temperature muffle furnaces that can sustain temperatures between 300 and 400 °C are used during the processes. In the presence of oxygen in the air, certain substances, like water and other volatile elements, are combusted while others, including organic molecules, are evaporated. The amount of ash in the sample can be calculated by comparing the weights of the sample before and after ashing. Fixed carbon is calculated by subtracting the percentage of volatile matter, moister content, and ash content from 100. The bulk density of the orange peel AC was determined with the help of a densitometer (BTI Make: Microprocessor Based 4 Digit present counter, 30 strokes per minute, height 50 mm). The chemical compositions of the orange peel AC were analyzed with energy-dispersive X-ray (EDAX) analysis shown in Figure 2. EDAX analysis of orange peel AC has the element consisting of C, O, Zn, etc. The physicochemical characteristics of the orange peel AC are presented in Table 1.

XRD analysis result

An X-ray diffractometer with Cu*K* as the radiation source was used to record the XRD profile of the prepared orange peels AC over the 2θ range of 10°–80°. In accordance with the findings of the XRD investigation, the diffractogram that is shown in Figure 3 reveals both broad peaks and the absence of a sharp peak. The absence of a clear peak may be an indication that the materials have an amorphous structure. The appearance of broad peaks at 24° and 46°, however, indicated the development of a crystalline carbonaceous structure, which in turn improved the alignment of the layers. This finding, which also matches JCPDS card no. 75-2078, indicates the partially graphitic structure of the orange peel AC sample. It is related to the (002) graphitic plane that the sharp peak at 26.12 occurs.

Fourier transform infrared spectroscopy (FTIR) analysis result

Figure 4 shows the results of an FTIR spectroscopy analysis conducted on AC made from orange peels. The sample demonstrated four major absorption bands at 1,900–2,100 cm^{-1}, 1,200–1,300 cm^{-1}, 750–850 cm^{-1}, and 100–300 cm^{-1} for orange peels AC. There is a graphical representation showing what proportion of the functional groupings falls into each category. The carbon–oxygen-functional groups that are present on the surface of the AC are one of the most critical characteristics that control and determine the adsorption of MR by the AC. Analyses of AC obtained from orange peels show the presence of lactone functional groups with a frequency of 1,460.81 cm^{-1}, carbonyl groups with a frequency of 1,293.6 cm^{-1}, and aromatic C–H groups with a frequency of 845.63 cm^{-1}. Absorptive peaks at roughly 3,568 cm^{-1} were observed in the FTIR spectrum of $ZnCl_2$-impregnated orange peels, indicating the presence of hydroxyl groups. Results showed that $ZnCl_2$ activation resulted in

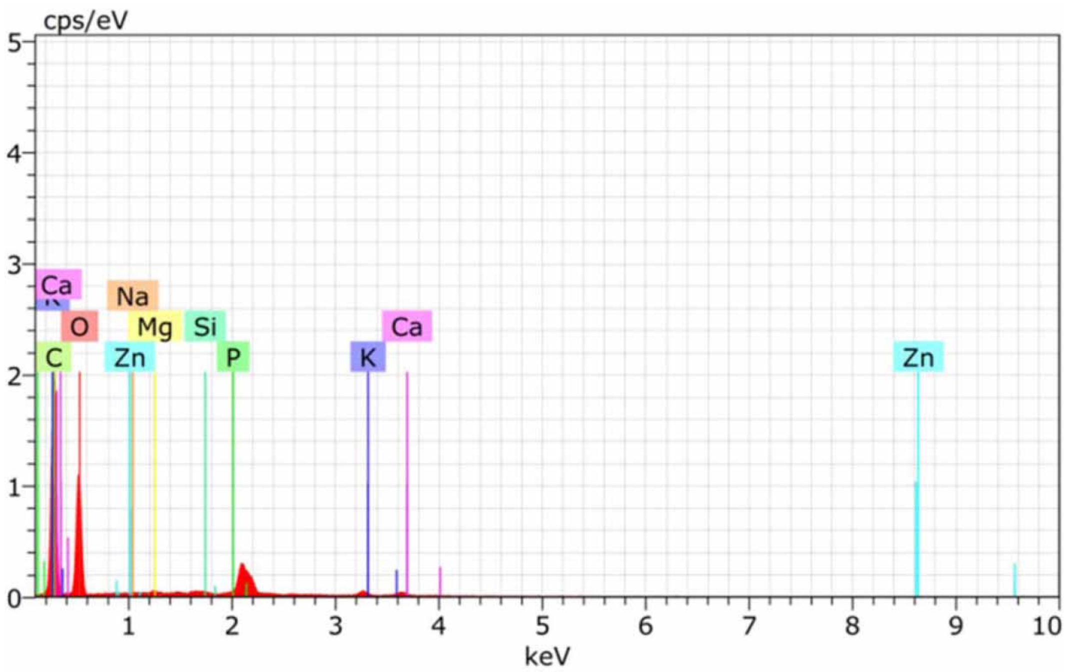


Figure 2 | EDAX of orange peels AC.

Table 1 | Characteristics of the orange peels AC

Properties	Units	Orange peels AC
Surface area	$m^2\ g^{-1}$	512.2
Total pore volume	$cm^3\ g^{-1}$	0.29
Average pore diameter	Å	33.21
Bulk density	g/cm^3	0.58
Moisture content	wt.%	8.19
Volatile matter content	wt.%	23.7
Fixed carbon content	wt.%	66.41
Ash content	wt.%	1.7

an oxygen-functional group-rich carbon surface. The intensity of the peak at 1,180 cm^{-1} of orange peel AC indicates that impregnation with $ZnCl_2$ leads to aromatic properties.

Scanning electron microscopy (SEM) analysis result

The pore structure of the orange peels AC that was obtained was analyzed using a scanning electron microscope (SEM; FEI Nova NanoSEM 450 Model). The SEM equipment has ultra-high-resolution low voltage imaging and unique low vacuum capabilities. The SEM images were taken in different resolutions 1 nm at 15 kV, 1.4 nm at 1 kV, 1.8 nm at 3 kV, and 30 Pa. Observing the surface physical morphology of the AC found in orange peel was accomplished through the use of the SEM technique. Figure 5 shows the SEM image of orange peel AC. It is an indication that the outside surface of the AC has macropores that are uniformly open. In addition, the surface of AC is highly porous and characterized by its unevenness. It is also clear that the orange peel AC has micropores and cavities on its outer surface, where the dark area resides. After being subjected to carbonization, orange peels AC develop voids (called pores) due to the evaporation of $ZnCl_2$. It is possible to think of these pores as channels leading to the microporous network.

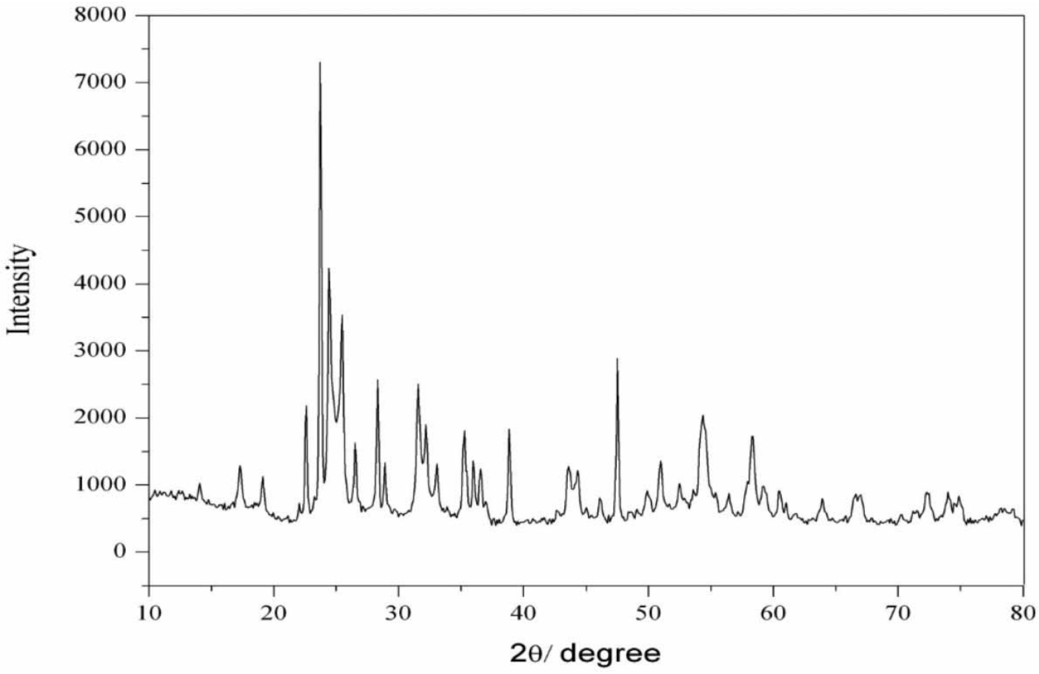


Figure 3 | X-ray diffractogram for orange peels AC.

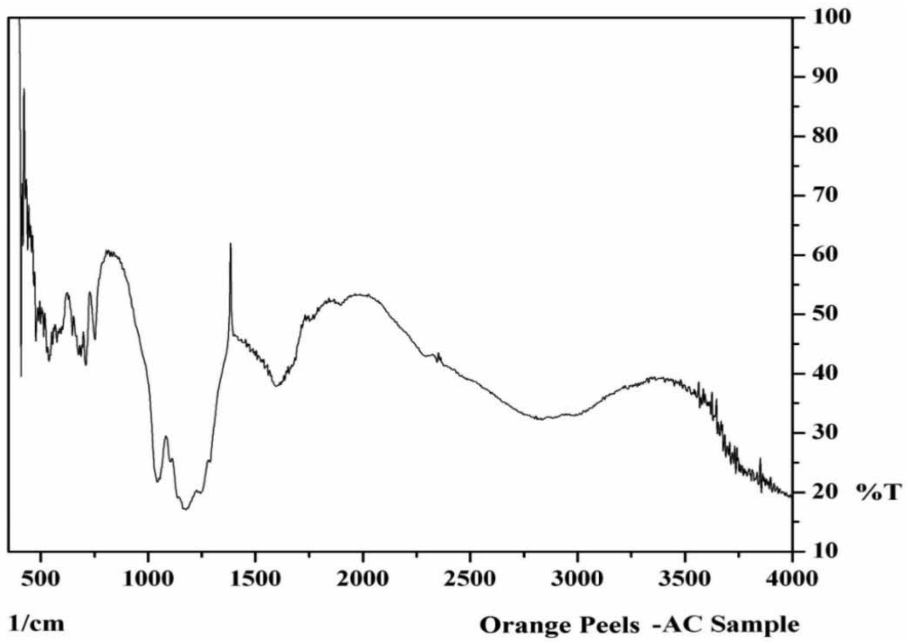


Figure 4 | FTIR for orange peels AC.

Studies on batch adsorption

Impact of initial MR concentrations

Dye adsorption onto AC made from orange peels was tested under various conditions to see how solution concentration at the outset affected the overall adsorption time. The studies were conducted at 60 °C and pH 7 with a constant adsorbent dose

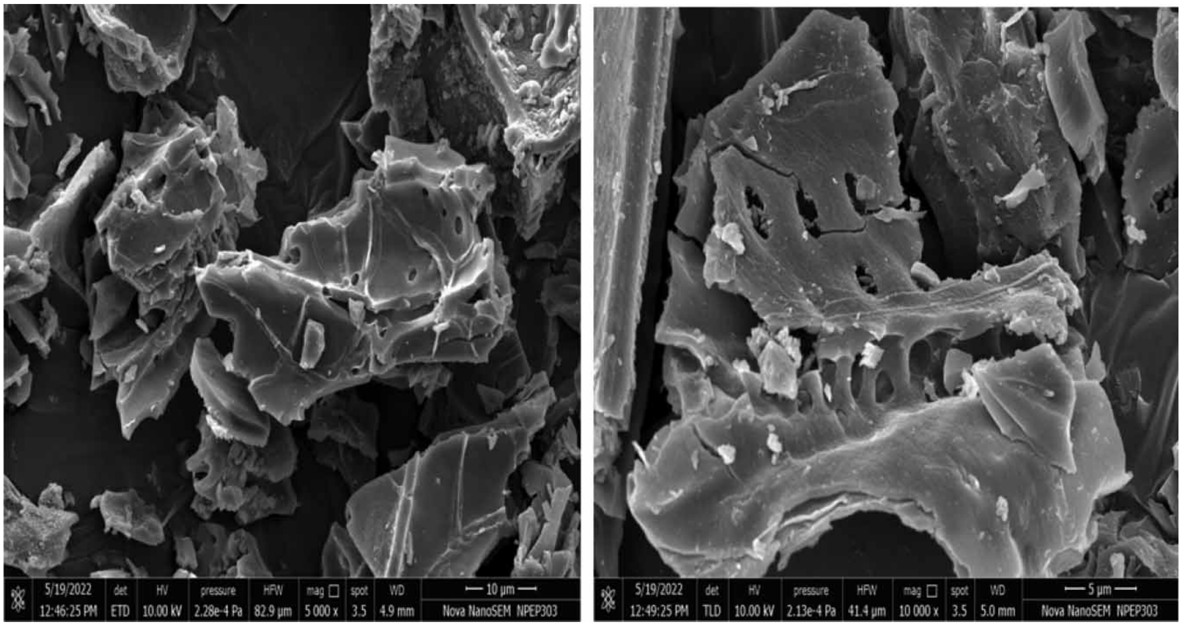


Figure 5 | SEM image of orange peels AC.

of 1 g/L and varying initial MR concentrations ranging from 100 to 400 mg/L. Dye uptake by orange peels AC is quick for the first 40 min, and then proceeds to a slower pace and eventually attains saturation, as shown in Figure 6, which displays the influence of varying initial concentrations on the percentage removal of MR. This could be due to quick adsorption occurring on the outside surface, followed by delayed adsorption occurring inside the material's pores. The equilibrium removal of dye for orange peels AC dropped from 100 to 400 mg/L when the initial MR concentration was raised. At low concentrations, almost all MR molecules were adsorbed on the surface, whereas higher quantities led to rapid saturation of orange peel AC surfaces.

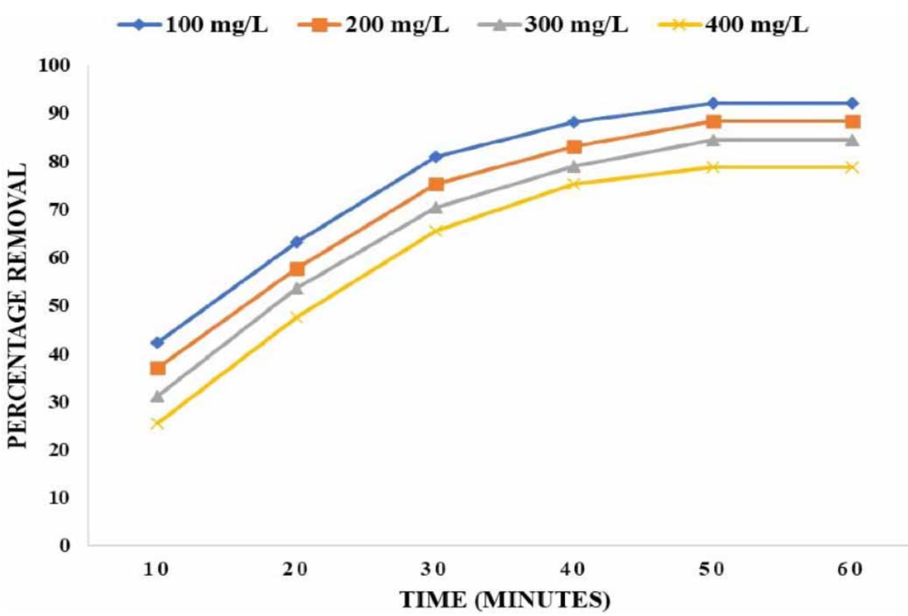


Figure 6 | Impact of initial MR concentration and contact time on orange peels AC.

Impact of orange peel AC (adsorbent) dosage

Adsorbent capacity for a given starting adsorbate concentration is determined by the dosage of the adsorbent; hence, this parameter is crucial. The effectiveness of different adsorbent doses in removing the MR using AC from orange peels was investigated, with the starting MR concentration held constant at 100 mg/L. Figure 7 depicts the influence of adsorbent dosages on the percentage of MR removal, and it is evident that the percentage of MR removal increased up to 1 g/L adsorbent dosage, after which it remained nearly constant. The increase in adsorption dosage leads to an increase in the available surface area, which increases the availability of additional sites for adsorption. The further increase causes the saturation of the adsorption sites. Therefore, it is self-evident that equilibrium was arrived at more quickly at lower concentrations. Because of this, AC from orange peels was chosen as the adsorbent at a dosage of 1 g/L for MR removal. At a 1 g/L adsorbent dosage, orange peel AC was used in all of the studies.

Impact of pH

Since wastewater that contains dyes is discharged at varying pH levels, it is essential to investigate the function that pH plays in the removal of MR. The pH of the MR solution impacts the adsorbent's surface charge, ionization, and dissociation of functional groups on active sites. Figure 8 illustrates the impact that the pH has on AC made from orange peels when the temperature is 60 °C, the MR concentration is 100 mg/L, and the 1 g/L adsorbent dose. Experiments were conducted with pH ranging from 3 to 11 to determine the pH's impact on the removal efficiency. However, dye uptake increases significantly from pH 7 to 9, and then slightly from pH 9 to 11. At a pH of 11, the removal of MR was found to be at its maximum.

Impact of temperature

Experiments on adsorption were performed at various temperatures (40–60 °C) and initial MR concentrations (100 mg/L) at a pH value of 7. Time rate experiments for MR adsorption onto orange peels AC at various temperatures (40–60 °C) are shown graphically in Figure 9 and it has been seen that when the temperature rises, the amount of MR that is removed by AC from orange peels increases in percentage. The MR percentage removal by orange peels AC from 89.28 to 92.78%. When the temperature is increased from 40 to 60 °C, there is almost little uptake of the MR. So, all batch mode studies were done at 60 °C.

Isotherms studies

When adsorption reaches equilibrium, the isotherm reflects how molecules are divided across the liquid and solid phases (Lopicic *et al.* 2021; Sivakumar *et al.* 2022). At a range of temperatures, a study of adsorption isotherms was conducted

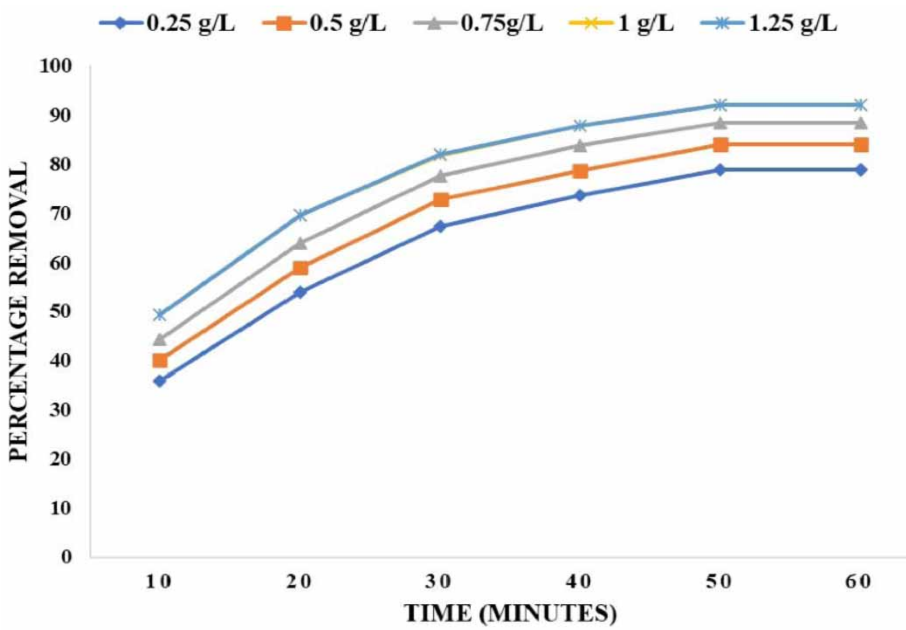


Figure 7 | Impact of adsorbent dose on percentage removal of MR using orange peels AC.

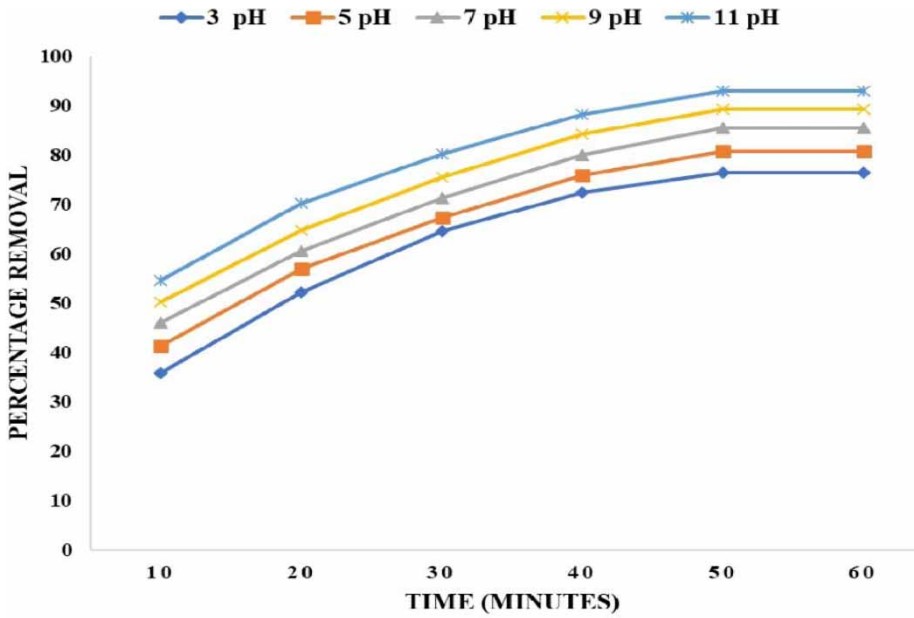


Figure 8 | Impact of pH on the percentage removal of MR onto orange peels AC.

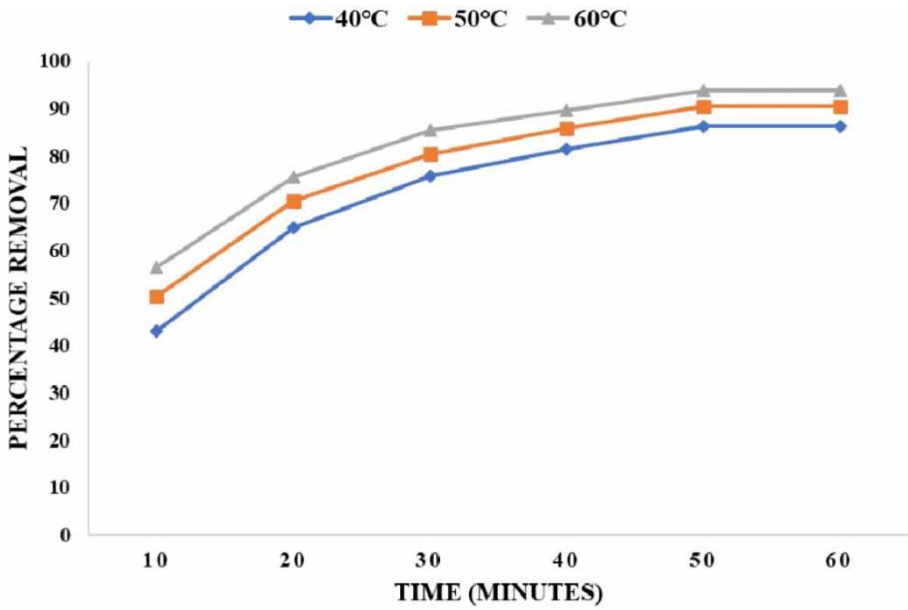


Figure 9 | Impact of temperature on the percentage removal of MR using orange peels AC.

using the well-known Langmuir and Freundlich isotherms (Babatunde *et al.* 2016). The Langmuir model is one that is utilized well in various monolayer adsorption procedures. This model operates under the presumption that adsorptions take place on the adsorbent at particular homogeneous spots. The Freundlich model acknowledges the existence of heterogeneity on the surface and makes the assumption that adsorption takes place at locations that have varying amounts of adsorption energy. The R^2 values of the correlation coefficients are used while assessing the value of the isotherm equation.

Langmuir isotherm

Researchers have used the Langmuir isotherm to predict the adsorption of different heavy metal ions, dyes, and organic pollutants onto AC (Gedam *et al.* 2019; Wong *et al.* 2019). The maximum adsorption capacity of an adsorbent can be calculated using the Langmuir isotherm model. This model assumes that the adsorbent has perfect monolayer coverage (Balarak *et al.* 2015). The isotherms can be studied by plotting the respective graphs based on the equilibrium. The Langmuir model was utilized in order to analyze the equilibrium data pertaining to the adsorption of MR onto AC made from orange peels. Figure 10 shows a linear relationship between the specific adsorption capacity (C_e/q_e) and the equilibrium concentration (C_e), with a coefficient of correlation (R^2) of greater than 0.96, confirming the viability of the Langmuir model and evidencing the homogeneous nature of the AC from orange peels. The slope in Figure 10 was found to achieve the value of $1/(q_{max}K_L)$. The value of $1/q_{max}$ came from the y-axis intercept. The results also showed that a monolayer of MR molecules formed on the adsorbent's surface. At a temperature of 60 °C, the Langmuir model predicted that the MR would have a maximum monolayer capacity of 111.11 mg/g when adsorbed onto AC from orange peels. Table 2 shows the Langmuir isotherm constants.

Freundlich isotherm

Dye adsorption on a heterogeneous surface is assumed not to be restricted to a monomolecular layer, leading to the proposed Freundlich model (Balarak *et al.* 2015). The Freundlich equation was used to examine equilibrium data for MR adsorption onto AC from orange peels. The straight line with an intercept of ln K_F and a slope of $1/n$ can be seen in the plot of ln q_e vs. ln C_e which is shown in Figure 11. The Freundlich model correlation coefficients (R^2) suggest that experimental data fitted to the model, and $n > 1$ indicates that dye adsorption onto orange peel AC is a favorable adsorption process. When compared with the Langmuir isotherm models, the R^2 value suggests that the Freundlich isotherm is less relevant. Table 3 shows the Freundlich isotherm constants.

Comparative study

After discussing all the process parameters, the results for orange peels AC were compared with the locally available commercial activated carbon (CAC). Figure 12 represents the comparative experimental data with CAC.

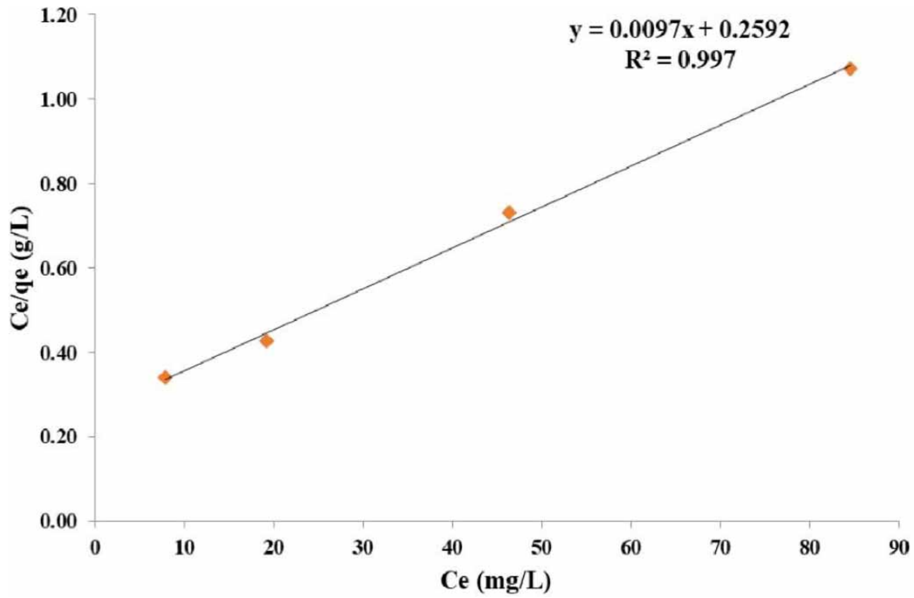


Figure 10 | Langmuir isotherm at 60 °C, 11 pH, 100 ppm, 1 g/L orange peels AC.

Table 2 | Langmuir isotherm constants

Temperature (°C)	Constants	Magnitude
60	K_L (L/mg)	0.033
	q_{max} (mg)	111.11
	R^2	0.997

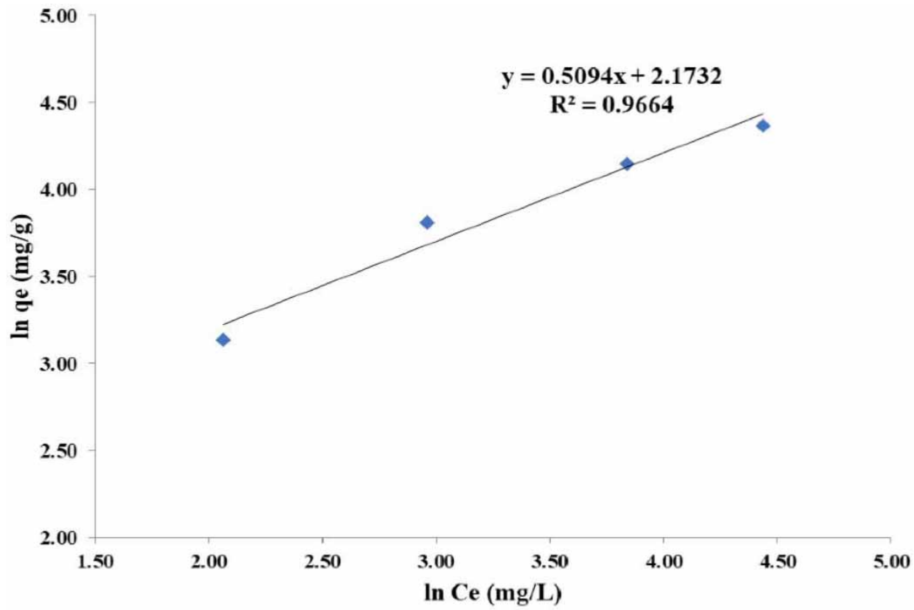


Figure 11 | Freundlich isotherm at 60 °C, 11 pH, 100 ppm, 1 g/L orange peels AC.

Table 3 | Freundlich isotherm constants

Temperature (°C)	Constants	Magnitude
60 °C	K_F (mg g^{-1}) (mg^3/g)$^{1/n}$	8.07
	n	1.96
	R^2	0.966

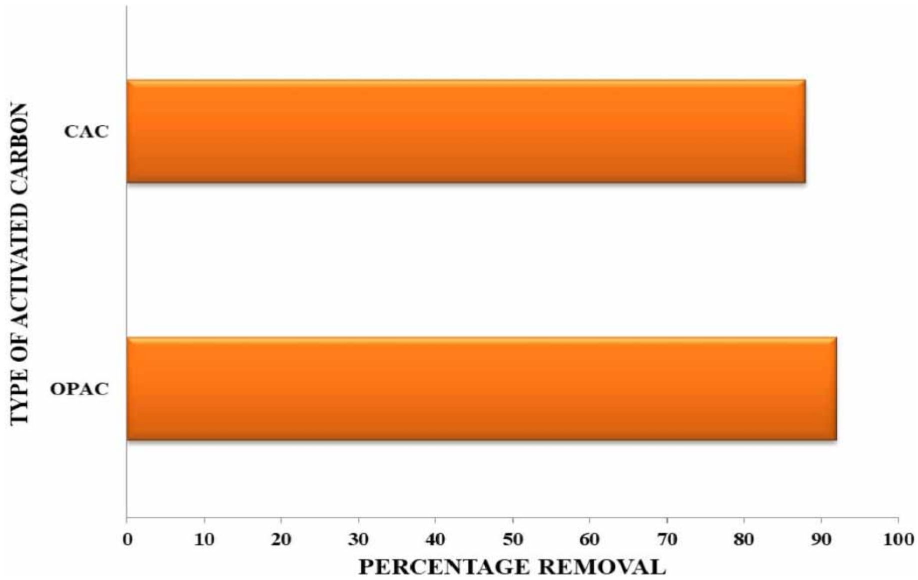


Figure 12 | Comparative data of orange peels AC vs. CAC at 60 °C, 11 pH, amount of adsorbent 1 g/L for 100 mg/L.

CONCLUSION

Methyl red (MR) adsorption onto orange peel AC has been investigated. The concentration of MR, the adsorbent dosage used, solution pH, and adsorption temperature are the primary variables affecting the efficiency of MR removal. Adsorption efficiency was observed to increase with increasing solution temperature (60 °C), contact time (60 min), and adsorbent dose (1 g/L), but to decrease with increasing initial dye concentration. Increases in pH led to more MR removal, with optimal adsorption achieved at pH 11. Langmuir and Freundlich's isotherms were used to fit equilibrium data, with Langmuir providing a better fit with 111.11 mg/g at 60 °C. The results of the study that compared orange peel AC with CAC demonstrate that orange peel AC provides a higher percentage removal of MR than CAC. According to the findings of this research, AC from orange peels is capable of functioning as a useful material for the removal of MR as an adsorbent.

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DATA AVAILABILITY STATEMENT

All relevant data are included in the paper or its Supplementary Information.

CONFLICT OF INTEREST

The authors declare there is no conflict.

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