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Adsorption of the Textile Dye RED 6 on Activated Carbons Derived from Neem Bark (*Azadirachta indica*)

Carine Emilienne Ouedraogo [a*], Ladji Meité [a], Coulibaly Yaya [b], Sory Karim Traore [a], and Koné Mamadou [a]

[a] Department of Sciences and Environmental Management, Laboratory of Environmental Sciences, NanguiAbrogoua University, 02 BP 801 Abidjan 02, Côte d'Ivoire.
[b] Department of Agro-Industrial Science and Technology, UFR Agriculture Halieutic Resources and Agro-industry, Polytechnic University of San Pedro, BP 1800 San Pedro, Côte d'Ivoire.

Authors' contributions

This work was carried out in collaboration among all authors. All authors read and approved the final manuscript.

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ABSTRACT

The elimination of dyes is a major challenge in our developing countries. The aim of this work is to demonstrate the effectiveness of activated carbon from local biomass in eliminating the textile dye RED 6 using adsorption. The activated carbons were prepared chemically using phosphoric acid (H3PO4) as the activating agent. RED 6 was prepared by dissolving the powdered dye in distilled water. To eliminate the RED 6 dye, a series of adsorption tests were carried out. The influences of contact time, dye concentration, mass, pH and temperature were determined and expressed as adsorption rate and adsorbed quantity. The preparation yielded activated carbons CA 30 and CA

**Corresponding author: E-mail: miss.carineemilienne@yahoo.fr;*

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60. The dye solution RED 6 with a concentration of 1 g/L was obtained. The optimum adsorption mass is 15 g for CA 60 carbon and 24 g for CA 30 carbon. The RED 6 dye is adsorbed in large quantities throughout the chosen pH range. These results show that the optimum concentration for both carbons is 100 mg/L. The influence of contact time indicates that the best adsorption occurs at 40 and 60 min for carbons CA 60 and CA 30 respectively. The results of the temperature study show better adsorption of the RED 6 dye, with an adsorbed quantity of 4.13 mg/g for temperatures of 30 and 35°C. All these results show that CA 60 and CA 30 carbons produced from neem bark adsorb the RED 6 dye effectively and can be used to eliminate coloured effluents.

Keywords: Adsorption; activated carbons CA 30 and CA 60; neem bark; Textile dye RED 6; phosphoric acid.

1. INTRODUCTION

Most countries in the world, particularly developing countries such as Côte d'Ivoire, are faced with the problem of water pollution. In fact, domestic and industrial effluents are discharged into watercourses via gutters. These effluents are generally highly coloured, with large quantities of suspended organic matter. By their very nature, synthetic dyes are recalcitrant to biodegradation, so biological treatment processes are ineffective in removing them from wastewater (Baban et al., 2010). Their high concentration in the aquatic environment causes a reduction in photosynthetic activity, resulting in the asphyxiation of animals and plants (Tamez et al., 2009). A number of studies have also highlighted the mutagenic and carcinogenic properties of many chemicals, especially dyes and their degradation products for humans and animals (Chen, 2006; Cuoco, 2009; Fayoud et al., 2015; Meroufel, 2015). This is why their treatment is a major concern for environmentalists and researchers.

The classic treatment consists of eliminating these pollutants, most of which are organic, by biological means, ozonation, etc. Unfortunately, it is difficult to eliminate most dyes because of their synthetic nature and the complexity of their structures, which include aromatic nuclei that considerably reduce their biodegradability (Aboua, 2013). Adsorption on activated carbons is an alternative method for solving this problem.

This technique uses highly porous materials from local biomass (wood, coconut, etc.) with a large specific surface area of up to 2,500m^2/g (Sun & Meunier, 2003; Morlay et al., 2006; Bendjelloul, 2017). Several studies have been carried out on dye adsorption on activated carbon produced from local biomass. The shells of moringa pods (Sanogo et al., 2020) and coffee pods (Kokora et al., 2020) were used respectively for the elimination of the dyes Green 42 and acid yellow 11 in an aqueous medium.

This study therefore proposes to eliminate the textile dye RED 6 by adsorption on activated carbon derived from neem bark. Neem's thick, hard bark is ideal for producing very good carbon. Adsorption tests will be carried out by varying various parameters (pH, mass, time, temperature and concentration).

2. MATERIALS AND METHODS

2.1 Materials

2.1.1 Solvent and equipment

The solvents used were analytical grade. The commercial solution of phosphoric acid (H_3PO_4) of 85% purity was supplied by the Fluka Company. It was used to impregnate neem bark. The HANNA HI 991001 pH meter was used to determine the pH of the solutions. The reactors were stirred using an RS-3 magnetic stirrer.

2.1.2 Red 6textile dye

RED 6 is a synthetic anionic dye in powder form. It is used to dye cotton and cellulose fibre fabrics, silk and printing wool. The choice of dye is based on how dangerous it is in the environment. This azo dye, which is highly soluble in water, causes a number of illnesses including cancer and genetic mutations (Chen, 2006).

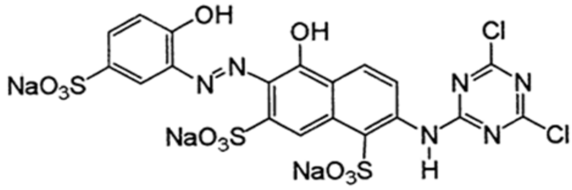
Table 1 shows the different characteristics of the RED 6 dye.

2.2 Methods

2.2.1 Preparation of activated carbon

The protocol used for the preparation of activated carbons (AC) is that of authors (Ouedraogo et al., 2021; Gueye et al., 2011) who produced activated carbons from plant biomass. After acquiring the biological material, the bark was washed with plenty of tap water to remove impurities such as sand and dust.

Table 1. RED 6 dye characteristics

Usual name	**RED 6 or Rubine B**
Structure and systematic name	 disodium salt of 3-hydroxy-4-[(4-methyl-2-sulfophenyl)azo]-2-naphthalenecarboxylic acid
Molar mass	733,38 g/mol
Molecular formula	$C_{19}H_9Cl_2N_6Na_3O_{11}S_3$
wavelength	552 nm
Purity	100 %

Then the barks were dried in an oven at 110 °C for 24 h before the production of the activated carbons by the chemical method. The carbons were prepared by impregnating, in reactors, 150 g of neem bark with 150 mL of phosphoric acid (H_3PO_4) at concentrations of 30% to 60%, with stirring, for 24 h. The matter obtained was dried at 110°C/24h and then calcined at 550 °C for 1h30min. Then the resulting carbons were washed, dried, crushed and finally sieved to have a diameter of between 2 and 2.4 mm which was used for the rest of the work.

2.2.2 Preparation of RED 6 dye

RED 6 dye solution was prepared by dissolving 1 g of dye powder in 1 L of boiling distilled water. This produced a stock solution with a concentration of 1 g/L. This solution was diluted to produce daughter solutions with concentrations ranging from 25 mg/L to 150 mg/L. These solutions were used to plot the calibration curve shown in Fig. 1.

2.2.3 Adsorption tests

The various tests were carried out to determine the influence of various parameters such as the contact time, the initial concentration of the RED 6 dye, the mass of carbon, the pH of the RED 6 solution and the temperature of the reaction medium. The following protocol was adopted for the adsorption tests: the conditions were set and each parameter studied was varied. For each test, a known mass of charcoal was introduced into a fixed volume of dye solution of known concentration. The mixture was agitated and samples were taken at regular intervals. The sampled solutions were centrifuged and then analysed using a UV/Visible spectrophotometer to measure the residual concentration of the dye.

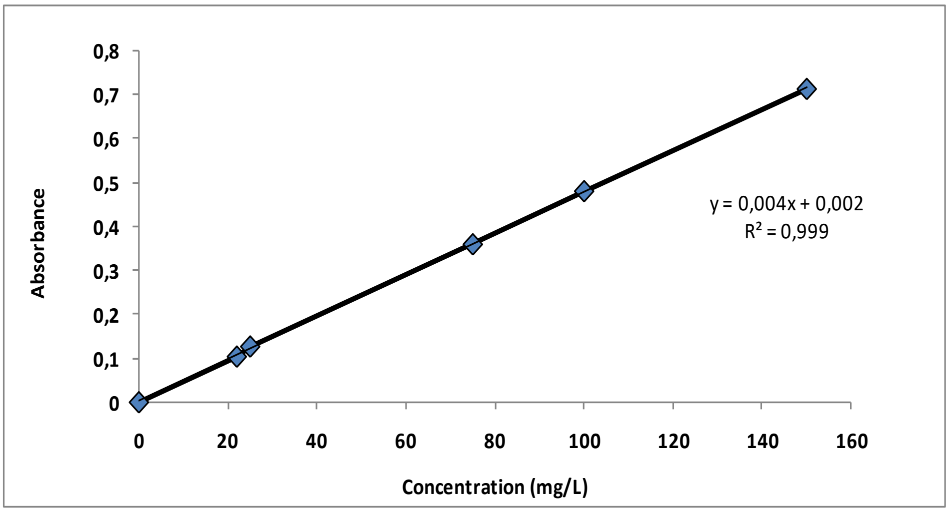


Fig. 1. Calibration curve for RED 6 dye

Parameter variations were carried out at times ranging from 10 min to 120 min, masses from 10 g to 30 g, concentrations from 25 mg/L to 150 mg/L, pH from 2 to 10 and temperatures between 30°C and 50°C.

2.2.4 Calculation methods

For the purposes of this study, the results are expressed either as the quantity adsorbed or as the adsorption rate. Each residual concentration obtained is measured in triplicate in order to obtain an average value for this concentration. The quantity of dye adsorbed expressed in mg of solute per gram of solid adsorbent is given by relationship (1):

$$Q_t = \frac{(C_i - C_t)}{m} \times V (1)$$

- Q_t: quantity of micropollutant per unit mass of carbon (mg/g);
- C_i: initial concentration (mg/L);
- C_t:residualequilibrium concentration (mg/L);
- m: mass of carbon (g);
- V: volume of solute (L).

The calculation of the adsorption rate expressed as a percentage is given by formula (2):

$$Taux\ d'adsorption(\%) = \frac{C_0 - C_1}{C_0} \times 100 (2)$$

- C_0: initial concentration (mg/L);
- C_1: residual equilibrium concentration (mg/L).

3. RESULTS AND DISCUSSION

3.1 Influence of Contact Time

The results obtained for activated carbons with 60% (CA 60) and 30% (CA 30) phosphoric acid are shown in Fig. 2.

The results of the kinetic study illustrated in Fig. 2 show 2 very distinct parts: rapid adsorption up to the optimum time of 40 min for activated carbon with 60% phosphoric acid (CA 60) with an adsorbed quantity of 1.72 mg/g and 60 min for activated carbon with 30% phosphoric acid (CA 30) with an adsorbed quantity of 1.17 mg/g. After these optimum times,a slowdown in adsorption was observed, leading to a plateau.

The quantity of pores available on the surface allows an increase in the quantity of carbon adsorbed at the start of adsorption. In addition, pore saturation after 40 and 60 minutes respectively for CA 60 and CA 30 carbons leads to a plateau despite a prolonged contact time.These results are in line with those of (Abbaz et al., 2014; Enaime et al., 2017; Alcaraz et al., 2018).

For them, the very high quantity adsorbed at the beginning can be explained by the fact that, initially, the pores (micro and mesopores) are vacant and therefore easily accessible to the dye particles. However, once equilibrium is reached, adsorption becomes less pronounced and more stable, due to slower diffusion of dissolved species through the pores of the carbon.

3.2 Influence of concentration

Fig. 3 shows the results for the quantity adsorbed as a function of time for each concentration.

Fig. 3 shows that increasing the initial concentration leads to an increase in the quantity adsorbed on the two carbons. In fact, the quantity adsorbed increased from 0.95 to 2.89 mg/g for CA 60 carbon and from 0.6 to 1.43 mg/g for CA 30 carbon for RED 6 dye concentrations ranging from 25 to 150 mg/g. Furthermore, the quantity adsorbed hardly changes between concentrations of 100 mg/L and 150 mg/L.

This phenomenon could be explained by the fact that as the dye concentration increases, the pores available on the surface of the carbons remain constant. These saturated pores (micro and mesopores) have difficulty in adsorbing the RED 6 dye molecules. Similar results were obtained by authors (Enaime et al., 2017; Alcaraz et al., 2018) who studied the adsorption of dye on activated carbon from agricultural waste. They explain this result by the fact that increasing the initial concentration leads to an increase in the number of moles of dye, while the specific surface area of the two carbons remains constant.

3.3 Influence of the Mass of Activated Carbon

The results are illustrated in Fig. 4, which shows the adsorption rate as a function of the different masses.

The observation made from this figure is that, in general, the adsorption of the dye RED 6 is improved as the mass of carbon increases.

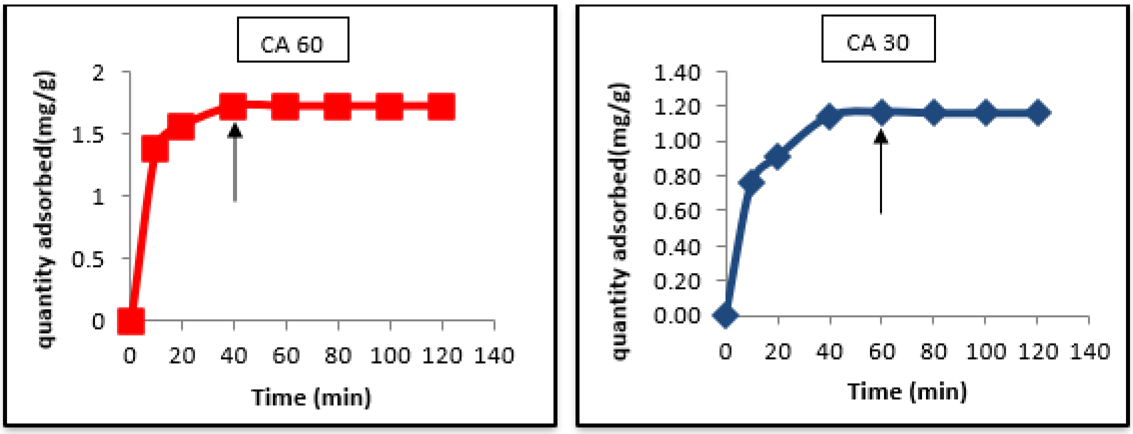


Fig. 2. Quantity of Red 6 dye adsorbed (mg/g of carbon) as a function of time

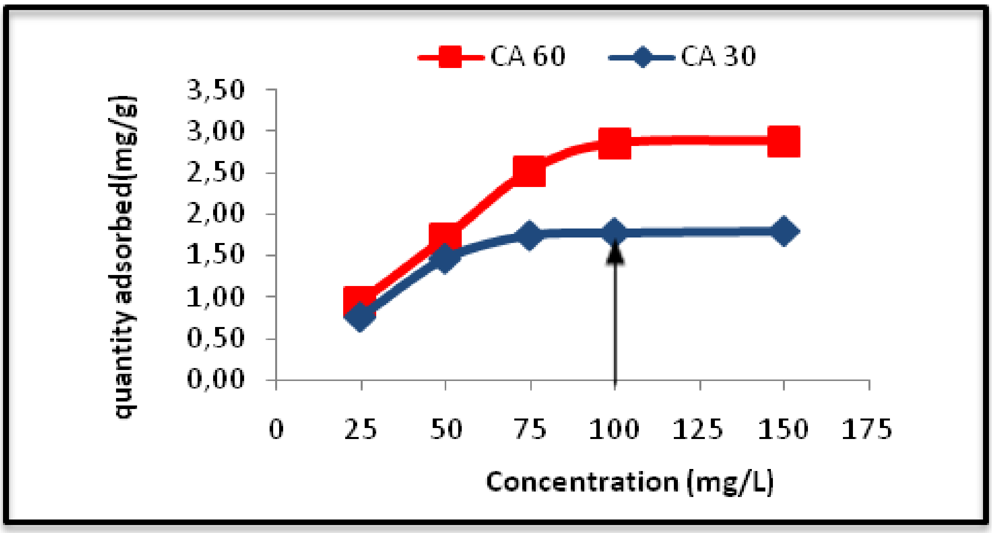


Fig. 3. Quantity of RED 6 adsorbed as a function of concentration

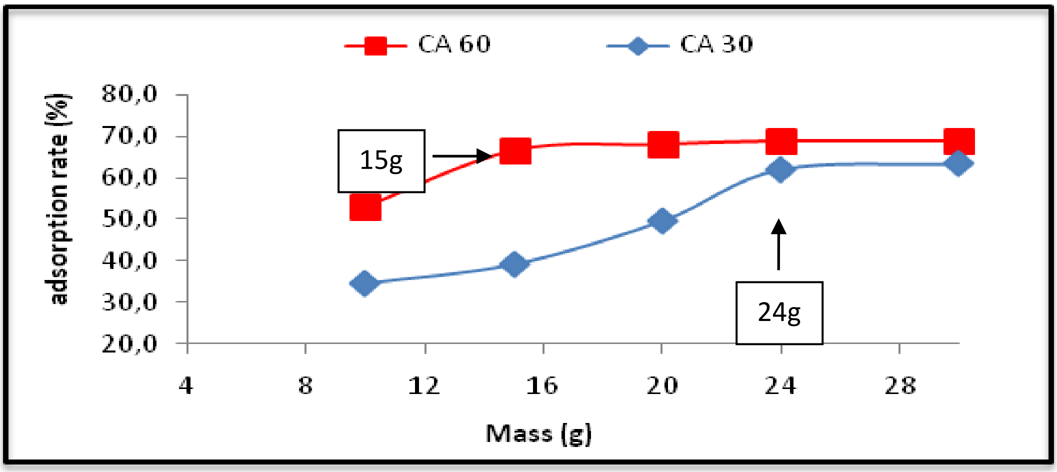


Fig. 4. Adsorption rate of RED 6 dye as a function of carbon mass

There is a rapid increase in the rate of adsorption when the mass increases from 10 to 15 g for CA 60 carbon and from 10 to 24 g for CA 30 carbon.The adsorption rate rises from 53% to 66.67% and from 34.67% to 62.00% respectively for CA 60 and CA 30 carbons. Above 15 and 24 g, the adsorption rate slows down, almost reaching a plateau.

The rapid adsorption is explained by the fact that increasing the mass of carbon leads to an increase in the overall contact surface area. This means that several adsorption sites are available to ensure greater retention of the RED 6 dye. Similar results have been obtained from work on the elimination of dyes from activated carbons (Reffas, 2010; Nebaghe, 2016; Seghier, 2018; El-Sayed, 2011). It should also be noted that, whatever the mass of activated carbon, the adsorption rate observed with CA 60 is relatively higher than that observed with CA 30. This could be explained by the high concentration of phosphoric acid used for activation. The mesoporous volume develops, resulting in greater adsorption of the dye. The subsequent stabilisation of the adsorption rate is due to saturation of the pores on the surface of the carbon. The addition of carbon increases the number of pores, but these additional additions lead to the formation of agglomerations of carbon particles causing pore obstruction (Chen et al., 2010).

3.4 Influence of the pH of the RED 6 Dye Solution

Fig. 5 shows the different pH adsorption rates as a function of time.

High retention of the RED 6 dye by the two carbons was observed over the entire pH range selected. This high retention is reflected in the high adsorption rates, with values in excess of 70%.

This result could be explained by the high concentration and mobility of OH- ions, which favours their adsorption to the detriment of the anionic dye. These results are supported by those obtained by (Elbariji et al., 2006) when removing the basic yellow XGL 250%. Authors (Aboua et al., 2015) also made the same observation when methylene blue was adsorbed onto coconut shell charcoal. In general, anionic dyes adsorb better in acidic media, which is due to the preponderance of positive charges.

3.5 Influence of temperature

The results of the study of the influence of temperature on the adsorption of the dye RED 6 by the carbons CA 60 and CA 30 are shown in Fig. 6.

Examination of Fig. 6 on the effect of temperature on the adsorption of CA 60 and CA 30 carbons shows that the quantity adsorbed remains stable at temperatures of 30 and 35°C and drops off at 40°C and above. The quantity adsorbed fell from 4.13 to 3.63 mg/g for CA 60 carbon and from 4.13 to 3.23 mg/g for CA 30 carbon. Generally speaking, when the temperature of the medium increases, this leads to a drop in the quantity of Red 6 dye adsorbed.

These results are in agreement with those of several authors (Belaid & Kacha, 2011; Gao et al., 2013). For the latter, this phenomenon is consistent with Arrhenius' law, which suggests that the surface reaction is

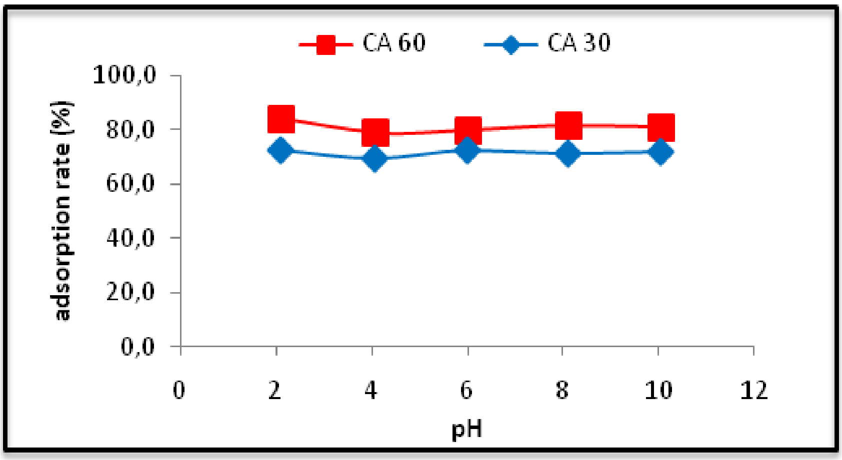


Fig. 5. Adsorption rate of Red 6 dye as a function of pH

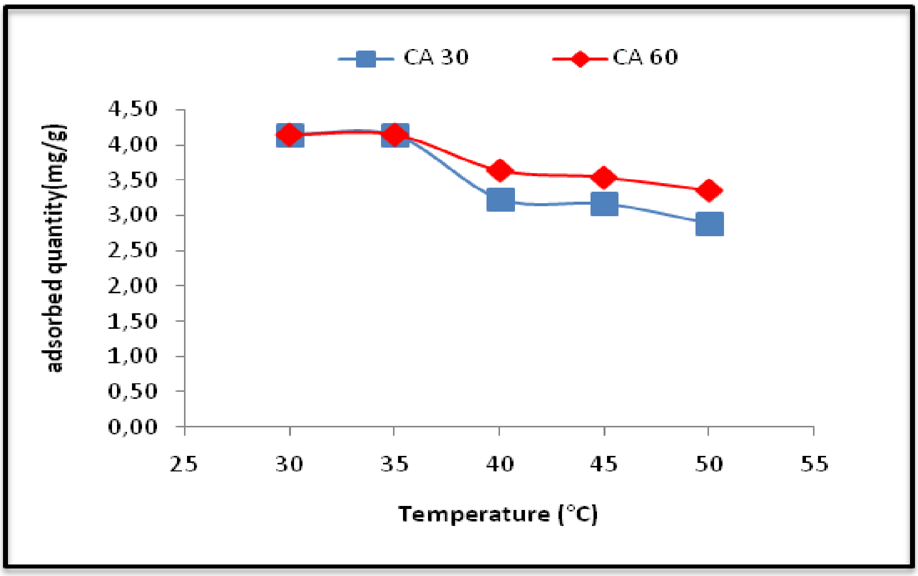


Fig. 6. Quantity of RED 6 dye adsorbed as a function of temperature

exothermic and that each increase in temperature hinders its progress.

4. CONCLUSION

The aim of this study was to eliminate the RED 6 dye using the adsorption method on activated carbon derived from neem bark. The various adsorption tests in static mode revealed that the optimum adsorption mass is 15 g for CA 60 carbon and 24 g for CA 30 carbon. The results of the pH variation indicate a high adsorption of the dye RED 6 over the entire pH range studied. Increasing the initial concentration leads to an increase in the quantity adsorbed until 100 mg/L is reached, at which point adsorption becomes slow and constant. The results of the contact time study indicate that the best adsorption occurs after 40 min for CA 60 and 60 min for CA 30. The study of temperature variation indicates that adsorption of RED 6 is effective at 30 and 35°C. The results show that adsorption on activated carbon from neem bark eliminates the textile dye RED 6 perfectly. This carbon could be used to eliminate other textile dyes or coloured effluents.

DISCLAIMER (ARTIFICIAL INTELLIGENCE)

Author(s) hereby declare that NO generative AI technologies such as Large Language Models (ChatGPT, COPILOT, etc) and text-to-image generators have been used during writing or editing of this manuscript.

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COMPETING INTERESTS

Authors have declared that no competing interests exist.

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