Solvent effect on the absorption and emission spectra of carbon dots: evaluation of ground and excited state dipole moment

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Abstract

Background: Carbon dots (C-dots) are photoluminescent nanoparticles with less than 10 nm in size. Today, many studies are performed to exploit the photoluminescence (PL) property of carbon dots, and our focus in this study is to estimate the dipole moment of carbon dots. For reaching our aims, C-dots were synthesized and dissolved in the different solvents.

Results: Carbon dots with intense photoluminescence properties have been synthesized by a one-step hydrothermal method from a carbon bio-source. In this research, we report on the effect of aprotic solvents on absorption and fluorescence spectra and dipole moments of C-dots dispersed in a range of many aprotic solvents with various polarity and dielectric constant at room temperature. The change in the value of dipole moment was estimated by using the Stokes shifts. The difference between the dipole moment of the excited state and the ground state was shown using an extended form of Lippert equations by Kawski and co-workers.

Conclusions: The values found for \( \mu_g = 1.077 \) D, and \( \mu_e = 3.157 \) D, as well as the change in the dipole moments. The results showed that the dipole moment of the excited state is more than the ground state, indicating a high density and redistribution of electrons in the excited state. Finally, the quantum yield of C-dots in the eclectic aprotic solvents was communicated and discussed.

Keywords: Carbon dots, Photoluminescence, Dipole moment, Quantum yield, KAT parameters, Solvatochromic

Introduction

Carbon-based nanomaterials such as carbon nanotubes, fullerene and graphene have poor solubility in water and lack strong fluorescence in the visible area, limiting their applications [1]. These shortcomings can be addressed by carbon dots (C-dots) which are spherical carbon-based nanoparticles with a size of less than 10 nm [2]. C-dots are heavily fluorescent, non-blinking, water soluble, chemically stable and can be easily synthesized at low cost [3, 4]. Also, introduction of full-color fluorescent C-dots [5] is another advantage that can expand their application spectrum. C-dots was first discovered by electrophoretic purification of single-walled carbon nanotubes in 2004 [6]. In recent years, different materials and synthesis methods have been used to obtain C-dots. The synthesis approaches of C-dots can be classified into two categories: top-down and bottom-up methods including hydrothermal [7, 8], electrochemical oxidation [9], acidic oxidation [10], microwave [11, 12], and laser ablation [13].

Owing to their physicochemical properties, C-dots can take part in the chemiluminescence reaction as oxidants,
emitting species, energy acceptors of chemical reaction energy or even as catalyst involving in different chemiluminescence systems [14, 15]. For the first time, Shen and et al. fabricated chemiluminescence C-dots and used it to develop new class of CL nanosensors for the imaging Reactive oxygen species [16]. Also C-dots have gained widespread attention in recent years, especially in chemical censoring [17], biosensing [18], bioimaging [19], drug delivery [20], solar cells [21], light-emitting diode (LED) [22], and electrocatalysis [23].

C-dots are easily dispersed in protic and aprotic solvents due to carboxyl, hydroxyl, and carbonyl groups. The interaction between C-dots and solvent plays an essential role in the wavelength of photoluminescence emissions. In summary, no single theory can be used for a quantitative explanation of the effects of the environment on fluorescence. Explanation of these effects depends not only on polarity considerations but also on the structure of the C-dots and the types of chemical interactions it can experience with other near molecules. Kumar et al. [24] report on solvent-dependent spectroscopic study of fluorescent carbon nanoparticles in organic solvents. They have found that the absorption spectra of the nanoparticles were independent of solvent nature, while their photoluminescence spectra were considerably dependent on the solvent nature. The trends observed with solvent polarity follow the theory of general solvent effects, which may give the impression that solvent polarity is the only factor to consider. Solvent–solute interaction and the trace of solvent environments are investigated by considering various solvent parameters such as hydrogen bond capability, hydrogen bond acceptability and polarization on dipole moment [25, 26]. Determining the dipole moment of electron balances is crucial because it can explain how electron distribution changes under excitation. Suppan has shown that the most acceptable method to approximate the excited dipole moment of a solute involves the simultaneous estimation of the absorption and fluorescence spectra of the solute in the range of solvent [27]. C-dot fluorescence properties are complicated by dependence on excitation wavelength [28] and solvent nature [24, 29]. Because there are functional groups on the C-dots surface, they are therefore available to solvent molecules so that strong interactions of C-dots with solvent molecules can have a significant effect on fluorescence. We use the solvatochromic method in which the amount of polarity of the base and dipole moment was calculated, followed by the investigation of their differences in a variety of aprotic solvents.

Different methods [28, 30–34] have been introduced for solvatochromic measurement of the dipole moment.

Pursuing our previous work on protic solvents [35], we aim to understand the effect of intermolecular interactions of synthesized C-dots and aprotic solvents. The focus is to study the spectral changes of C-dots in aprotic solvents by using the concept of Kamlet-Abboud-Taft’s linear solubility energy. Kamlet-Abboud-Taft’s equation is one of the most reliable methods of measuring solvent effects on dissolved C-dots. This equation applies the solvent polarity parameter result on the solute’s spectral features [36].

### Experimental

#### Materials

C-dots were synthesized by hydrothermal treatment of persimmon peel. All the solvents used in this research were of the highest degree of purity available from Merck. The physical properties and polarity functions of the solvents are given in Table 1; Spectroscopic polarity parameters in various aprotic solvents are provided in Table 2.

#### Synthesis and characterization of C-dots

The green C-dots synthesis method, their characterization and structures, were thoroughly described in our previous work [38].

Briefly, the C-dots were synthesized from persimmon peels by hydrothermal treatment. In the first step, persimmon was cut into pieces and ground into a mixture. Then, 50 mL ultrapure water was added, and the solution

### Table 1: Physical properties, and polarity functions in aprotic solvents

| Solvents    | ε     | n     | n*    | α    | β     | QY   |
|-------------|-------|-------|-------|------|-------|------|
| DMSO        | 47.24 | 1.47  | 1     | 0    | 0.76  | 0.26 |
| Acetonitrile| 36.64 | 1.34  | 0.75  | 0.19 | 0.4   | 0.12 |
| DMF         | 38.25 | 1.43  | 0.88  | 0     | 0.69  | 0.17 |
| DCM         | 9.1   | 1.42  | 0.82  | 0.13 | 0.1   | 0.12 |
| Acetic Acid | 6.15  | 1.37  | 0.64  | 1.12 | 0.45  | ND   |
| Diethyl ether| 4.33 | 1.34  | 0.27  | 0    | 0.47  | 0.53 |
| Dioxane     | 2.3   | 1.42  | 0.55  | 0    | 0.37  | 0.10 |
was kept for 15 min under magnetic stirring, and the obtained juice was autoclaved using a Teflon lined stainless steel autoclave reactor at 120 °C for 150 min. The autoclave was allowed to cool at room temperature, and the resultant dark brown solution was centrifuged at 10,000 rpm for 20 min to separate the larger particles. In the next step, the pH of the aqueous solution was adjusted to neutral with 1 M NaOH, and the C-dots solution was filtrated with a 0.22 μm filter membrane. In the final step, the C-dots solution was further purified by dialysis against (1000MWCO) deionized water for 24 h. The powder of C-dots was obtained by lyophilization for 48 h and stored at 4 °C until further use. The formation of C-dots with an average size of 2 nm was obtained.

Together with elemental analysis by CHN-analyser and using FE-SEM, the nitrogen and carbonyl-containing functional groups on the surface of C-dots were also revealed by the FTIR method. Accordingly, for further spectroscopic analyses, a 0.1% (W/V) C-dots solution was prepared in aprotic solvents by mixing for 4 h to obtain homogenous solutions.

**Absorption and emission spectroscopy**

UV–Vis absorption spectra of the C-dot solutions in different aprotic solvents were recorded by a CECIL CE7250 Spectrophotometer with a 1 cm quartz cuvette at room temperature over a wavelength range of 200–600 nm. Meanwhile, photoluminescence (PL) measurements were undertaken using a Cytation 5/Biotek/USA fluorescence spectrophotometer with excitation slit set at 1 nm pass and emission at 1 nm bandpass in 96 cell plates.

**Results and discussion**

The theory of universal solvent effects provides beneficial information for consideration of solvent-dependent spectral shifts. In explaining general solvent effects, the C-dots is a dipole in a continuous medium of uniform dielectric constant.

| Solvents        | νa (kK) = 10^3 cm⁻¹ | νf (kK) | νa – νf | νa + νf | f(ε,n)   | f(ε,n) + 2 g(n) |
|-----------------|---------------------|--------|--------|--------|----------|----------------|
| DMSO            | 35.77               | 21.92  | 13.84  | 57.70  | 0.841    | 1.660          |
| Acetonitrile    | 35.71               | 22.19  | 13.51  | 57.91  | 0.862    | 1.533          |
| DMF             | 35.21               | 22.22  | 12.98  | 57.43  | 0.839    | 1.606          |
| DCM             | 35.58               | 22.47  | 13.11  | 58.05  | 0.597    | 1.353          |
| Acetic Acid     | 35.46               | 22.57  | 12.88  | 58.03  | 0.497    | 1.200          |
| Diethyl ether   | 36.49               | 22.72  | 13.76  | 59.22  | 0.382    | 1.054          |
| Dioxane         | 35.58               | 22.88  | 12.70  | 58.47  | 0.045    | 0.801          |
Depending on the solvent’s polarity, the aprotic solvent used in the registration of UV–Vis absorption and emission fluorescence spectra of the C-dots, depending on the solvent’s polarity, influenced the positions, intensity, and shape of the solvent-C-dots complex. The UV–vis absorption spectra of the C-dots were observed in the UV region with maximum absorption at 237–256 nm and a tail extending into the visible range (Fig. 1). This is attributed to the n–π* transition of C=O band and π–π* transition of C=C band.

The behavioural mechanism of PL is not yet fully understood, and our recent studies in calculating the ground and excited state dipole moment are a step forward in understanding the mechanism of this C-dots effect. One possible reason for the PL behaviour is the presence of different particle sizes of C-dots; and the different distribution of C-dots surface energy traps, the nature of the surface, and the presence of numerous functional groups on the surface of the C-dots may result in a series of emissive traps between π and π* of C–C.

Estimation of the ground state and excited state dipole moments
In order to approximate the ground state and excited state dipole moments of the C-dots, spectral shifts (υ_A – υ_F) and (υ_A + υ_F) of fluorescence C-dots were calculated along with solvent polarity (υ_A and υ_F are the wavenumbers (cm\(^{-1}\)) of the absorption and emission).

The result demonstrates that the excited dipole moment is larger during the C-dots electronic transition than the ground dipole moment, i.e. \(\mu_e > \mu_g\). Therefore, the dipolar solvent polarisation, the Franck–Condon excited state, is more solvated foremost to the experiential redshift in the spectrum.

To a first estimate, this energy difference (in cm\(^{-1}\)) is a property of the refractive index (n) and dielectric constant (ε) of the solvent, and is described by the Lippert-Mataga [30, 31] equation as below:

\[
\begin{align*}
\varphi_A - \varphi_F &= \frac{2}{\hbar c} \left( \left( \frac{\varepsilon - 1}{2\varepsilon + 1} - \frac{n^2 - 1}{2n^2 + 1} \right) \left( \mu_e - \mu_g \right)^2 + \text{const} \right) \\
\varphi_A + \varphi_F &= -m_2 \left[ f(\varepsilon n) + 2g(n) \right] + \text{const}
\end{align*}
\]

(1)

In this equation \(h = 6.6256 \times 10^{-27}\) ergs is Planck’s constant, \(c = 2.9979 \times 10^{10}\) cm/s is the speed of light, and \(a\) is the radius of the cavity in which the fluorophore resides. In this equation, the opposite effects of \(\varphi_A\) and \(\varphi_F\) on the Stokes shift are significant. As the refractive index (n) increases, this energy difference decreases, whereas an increase in ε results in a larger difference between \(\varphi_A\) and \(\varphi_F\). The refractive index is a rapid frequency response that depends on the motion of electrons in solvent molecules that occur when light is absorbed. In contrast with the refractive index, the dielectric constant is a static and steady feature that depends on the electrons and molecular motions of the solvents’ organization around the excited state. Increasing the refractive index (n) of the ground and excited states is quickly stabilized by the motion of electrons in solvent molecules. This redistribution of electrons reduces the energy difference between ground and excited states. Lippert-Mataga framework, there is no consideration of specific interaction with solvent. Thus, several investigators attempted to extend and modify the Lippert equation. Kawski and co-workers [38–40] obtained a simple quantum mechanical second-order perturbation theory for absorption (υ_A) and fluorescence (υ_F) band shifts. By variation of ε and n in solvents, as explained below, functions \(f(\varepsilon, n)\) and \(g(n)\) refer to Bakhshiev [41] and Kawski-Chamma-Viallet [33, 34] relations, respectively. Consequentially the solvent dependent changes for the difference and sum of \(\varphi_A\) with \(\varphi_F\) have been defined by the following equations:

\[
\begin{align*}
\varphi_A - \varphi_F &= m_1 f(\varepsilon n) + \text{const} \\
\varphi_A + \varphi_F &= -m_2 [f(\varepsilon n) + 2g(n)] + \text{const}
\end{align*}
\]

(2)

(3)

where:
The parameters $m_1$ and $m_2$ can be determined from absorption and fluorescence band shifts ($\nu_A - \nu_F$) and ($\nu_A + \nu_F$) using the following equations:

$$f(\varepsilon, n) = \frac{2n^2 + 1}{n^2 + 2} \left[ \frac{\varepsilon - 1}{2\varepsilon + 1} - \frac{n^2 - 1}{2n^2 + 1} \right]$$

$$g(n) = \frac{3}{2} \left[ \frac{n^4 - 1}{(n^2 + 2)} \right]$$

$$m_1 = \frac{2(\mu_e - \mu_g)^2}{\hbar c a^3}$$

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**Fig. 3** Plot of $\nu_A - \nu_F$ (kK) vs. $f(\varepsilon, n)$ for C-dots in the aprotic solvents: (1) DMSO, (2) Acetonitrile, (3) DMF, (4) DCM, (5) Acetic acid, (6) Diethyl ether, (7) Dioxane

**Fig. 4** Plot of $\nu_A + \nu_F$ (kK) vs. $f(\varepsilon, n) + 2g(n)$ for CDs in the aprotic solvents: (1) DMSO, (2) Acetonitrile, (3) DMF, (4) DCM, (5) Acetic acid, (6) Diethyl ether, (7) Dioxane
\[
m_2 = \frac{2(\mu_e^2 - \mu_g^2)}{\hbar c a^3}
\]

where \(\hbar\) is Planck constant and \(c\) is the velocity of light; \(\mu_e\) and \(\mu_g\) are the dipole moments in the ground and excited states; and \(a\) is Onsager radius of the C-dots, which was obtained from the molecular model where the molar volume was calculated by DFT-B3LYP/6-311++G(d,p) level of theory using Gaussian 0.3 W computer program. The value of the Onsager radius of the C-dots is estimated to be \(a = 3.97\) Å.

Since the slope \(m_1\) and \(m_2\) from the graph in Figs. 3 and 4, fill in this value in Eqs. 8 and 9 we have:

\[
\mu_g = \frac{m_2 - m_1}{2} \sqrt{\frac{\hbar c a^3}{2m_1}}
\]

\[
\mu_e = \frac{m_2 + m_1}{2} \sqrt{\frac{\hbar c a^3}{2m_1}}
\]

Thus, the ratio of dipole moments in excited state and ground state is given by:

\[
\frac{\mu_e}{\mu_g} = \frac{m_1 + m_2}{m_1 - m_2} \mu_g
\]

The value of Stokes shift varies between 12.70 and 13.84 kK. The values of the Stokes shifts are expressive of the charge transfer transition. The emission of C-dots dispersed in an aprotic solvent such as dioxane (\(ε = 2.3, α = 0\)) was recorded at 22.88 kK. While, in the aprotic solvents such as DMSO (\(ε = 47.27, α = 0\)), it was registered 21.92 kK. The larger dielectric constants result in a difference in Stokes shift of about 1.14 kK, indicating an increase in dipole moment in the excited state.

Table 3 Regression fits to solvatochromic polarity scales for stokes shift of C-dots

| Radius ‘\(r\)’ (Å) | \(\mu_g\) (D) | \(\mu_e\) (D) | \(\Delta\mu\) (D) | \(\mu_e/\mu_g\) | \(m_1\) (cm\(^{-1}\)) | \(m_2\) (cm\(^{-1}\)) |
|---------------------|----------------|----------------|------------------|----------------|------------------|------------------|
| 3.97                | 1.077          | 3.157          | 2.08             | 2.931          | 696.4            | 1417.8           |

Effect of aprotic solvent on the absorbance and fluorescence spectra

The typical fluorescence spectra and absorption spectra of C-dots in different aprotic solvents are shown in Figs. 2 and 3, respectively. The emission spectra of C-dots are broad, with shifts depending on the solvents. The large spectral shift is apparent in the fluorescence spectra compared to the absorption spectra. The smaller spectral shift in the absorption spectra than the emission spectra and the higher residence time for fluorescence indicate two phenomena. Firstly, the dipole moment of the excited state is greater than the ground state in all aprotic solvents studied. Secondly, the energy level of the first excited state, \(S_1\), is stabilized compared to the ground state, \(S_0\), by solvation with increasing the solvent polarity. These phenomena result in a redshift or bathochromic shift of the fluorescence.

The solvent effect was preserved within the outline of the linear solvation energy relationships (LSER) established by Kamlet-Taft [42] multivariate regression, in which each of specific and non-specific interactions has a linear contribution to the total solvation energy of solvent.

\[
E_T = A_0 + S\pi^* + a\alpha + b\beta
\]

Here the coefficients \(\pi^*\), \(\alpha\), and \(\beta\) are the Kamlet-Taft solvatochromic parameters (KAT) which have been developed for scaling the dipolarity/polarizability.
hydrogen-bond donor acidity and hydrogen-bond acceptor basicity of solvent, respectively [36, 43, 44]. The \( A_p, a, b \) and \( s \) are regression coefficients, quantity of the sensitivity of \( E_T \) values to the acidity, basicity and dipolarity/polarizability, respectively. The fit parameters are represented in Table 4. The emission spectra energies were obtained by using Eq. 11.

\[
E_T \left( \text{kCal.mol}^{-1} \right) = \frac{hcN_A}{\lambda_{\text{max}}(\text{nm})} = \frac{28591}{\lambda_{\text{max}}(\text{nm})} \tag{12}
\]

The molar electronic transition energy values, \( E_T \), of the C-dots in solvents were calculated using Eq. 12. The results related to KAT parameters in fluorescence show that the primary distribution in C-dots is related to polarization interactions. Also, in the as-prepared C-dot, the \( \pi^* \) and \( \beta \) sign are negative, indicating the ability to accept hydrogen bonding and polarization. For making the data in Table 4 reasonable and comparable, we have transformed the values in Table 5 into percentage contributions.

Also, in this study, the focus is on the solvatochromic effect of C-dots with solvents of low dielectric constants. Hence, the \( \pi^* \), \( \alpha \) and \( \beta \) in aprotic solvents are shown in Fig. 5. As it is indicated in Fig. 5, linear dependence could have resulted from specific and non-specific interactions. The emission maximum \( E_T \) diagram in \( \pi^* \) for the range of aprotic solvents are shown in Fig. 5a. A linear dependence result indicates reasonable linearity with \( r = 0.7 \) between \( E_T \) and \( \pi^* \) in the range of aprotic solvents. The reason for this significant deviation from the \( \alpha \) parameter can be explained by the use of aprotic solvents that have low acidity, and in most cases, \( \alpha \) was zero (Fig. 5a and b).

Table 4 Linear correlations found by plotting \( E_T \) versus \( \pi^* \), \( \alpha \) and \( \beta \) according to the KAT equation

| Sample | \( A_p \) | \( s \) | \( a \) | \( B \) |
|--------|--------|-------|------|------|
| C-dots | 66.75  | -2.85 | 0.11 | -1.41 |

Table 5 Percentage contribution of solvatochromic parameters in aprotic solvents

| KAT parameters | \( P_{\pi^*} (\%) \) | \( P_\alpha (\%) \) | \( P_\beta (\%) \) |
|----------------|---------------------|------------------|------------------|
| C-dots         | 65.21               | 2.51             | 32.26            |

The main contribution to solvatochromic is not related to the \( \alpha \) scale of solvent HBD acidity and \( \beta \) scale of solvent HBA basicity parameters of solvent. This relationship associates with parameter \( \pi^* \), which shows the polarity/polarization of the solvents, and the measure of the stability of a charge or dipole moment with a dielectric effect [45]. The results show that non-specific interaction (dipole–dipole) plays a significant role in solvatochromic of \( n-\pi^* \) transition from edge band/edge states of C-dots [44].

The quantum yield of the C-dots dissolved in aprotic solvents was defined at an excitation wavelength of 350 nm using the following equation (Eq. 13)

\[
Q_{\text{CDs}} = Q_R \cdot \frac{I_{\text{CDs}}}{I_R} \cdot \frac{A_l}{A_{\text{CDs}}} \cdot \frac{\eta^2_{\text{CDs}}}{\eta^2_R} \tag{13}
\]

where \( Q \) is the quantum yield, \( I \) is the intensity of fluorescence spectra, \( A \) is the absorbance at the excitation wavelength, and \( \eta \) is the refractive index (1.33) of the solvent. The molar electronic transition energy values, \( E_T \), of the C-dots was obtained in diethyl ether (with a quantum yield 0.5) in 0.1 M \( \text{H}_2\text{SO}_4 \) solution as the reference [46, 47]. The subscripts ‘CDs’ stands for carbon dots and ‘R’ for the reference are used in this equation. while the C-dots was dissolved in water (\( \eta = 1.33 \), as the reference solution to keep their absorption at minimum \( \approx 0.05 \) by comparing the integrated fluorescence intensities using the Eq. 13 at excitation wavelength of 350 nm and the quantum yield was determined. The quantum yield results calculated for C-dots are given in Table 1. According to the results, the highest amount of quantum yield among the studied aprotic solvents was obtained in diethyl ether (with a quantum yield of 0.53). This high quantum yield value can be attributed to the minimum value of AN in diethyl ether, among other aprotic solvents studied. To further explain this, it is assumed that the combination of other factors, such as the carbon core domain, affects emissions. However, solvatochromism deals with the effects of surface groups, and in the case of organic solvents, the effect of specific interactions such as hydrogen bond donors was minimal. Henceforth, if we limit our attention to the electron-donating and accepting characters of the constituents, the functional groups on the surface of C-dots, including quantum yield, are conserved in a wide range of solvents, and some of these solvents are potentially useful in biological applications. As the results illustrated in Table 1 the fluorescence efficiency in some of the solvent are significant. Nonetheless, they show no clear
trend. Consistent with our previous study [35], it was shown that the solvent with greater AN (the higher electron-accepting character can be seen from their α values) was a more effective quencher.

Conclusions

In summary, in this research work, we dissolved C-dots in various aprotic solvents to explore the specific solvation effects. A different factor is expressed on C-dots solvent-soluble interaction is generally controlled by the polarizability and basicity parameters. Studies were performed to calculate the dipole moment. The results showed that the excited state dipole moment is higher than the ground state. The results related to KAT parameters in fluorescence indicates the occurrence of a bathochromic shift of the fluorescence.

One of the grand challenges in studying C-dots for biological applications and bio-imaging is to increase the duration and intensity of C-dots fluorescence.Steps for more efficient use of C-dots in this field prompted us to evaluate quantum yields, and the resulted values indicated significant improvements of C-dot’s quantum yields in some aprotic solvents.

Abbreviations

C-dots: Carbon dots; PL: Photoluminescence; KAT: Kamelt-Taft; NaOH: Sodium hydroxide; DMSO: Dimethyl sulfoxide; DMF: Dimethylformamide; DCM: Dichloromethane; FE-SEM: Field emission scanning electron microscopy; FT-IR: Fourier transform infrared; LSER: Linear solvation energy relationships; HBA: Hydrogen bond acceptor; HBD: Hydrogen bond donor.

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Authors' contributions

PMJ Conceptualization, methodology, validation, formal analysis, investigation, data Curation, Writing—original draft, Writing—review & editing, software, visualization and was a major contributor in writing the manuscript; AA and RSA: Conceptualization, methodology, investigation, resources, data Curation, Writing—original draft, Writing—review & editing, supervision, project administration; MPM: Conceptualization, methodology, validation, resources, Writing—original draft, Writing—review & editing, supervision, K.J.G: Supervision, project administration, review & editing. AA and P.J wrote the main manuscript text and MP prepared figures. All authors read and approved the final manuscript. All authors.

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Declarations

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Consent for publication

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Competing interests
The authors declare that there is no conflict of interest regarding the publication of this paper.

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