

Synthesis and Characterisation of Silver–2-Phenylenediamine–Cyclodextrin Nanomaterials

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ABSTRACT

The spectral characteristics of 2-phenylenediamine (2PDA) in various solvents, and in the presence of α -cyclodextrin (α -CD) and β -cyclodextrin (β -CD) at pH~3, and pH ~7, were investigated using UV–visible, fluorescence, time-resolved fluorescence measurements, and PM3 computational methods. 2PDA showed a single broad emission band in all solvents, whereas dual emission observed in CD solutions indicates the presence of excimer in the 2PDA molecule. The fluorescence lifetimes of the inclusion complexes were greater than that of free 2PDA. In the 2PDA molecule, both the vertical and horizontal bond lengths between the both amino groups are smaller than the β -CD cavity size. The Ag:2PDA: α -CD and Ag:2PDA: β -CD nanomaterials were synthesized and characterized by SEM, FTIR, and DSC techniques. In all pH conditions, 2PDA exhibited distinct absorption and emission shifts upon complexation with α -CD and β -CD. SEM–EDX data confirmed the presence of 5.5% silver in the nanomaterials.

Keywords: 2-phenylenediamine, cyclodextrin, silver nano, pH effects, excimer, nanomaterials

INTRODUCTION

The ability of cyclodextrins (CDs) to accommodate guest molecules of suitable size within their cavities has been widely utilized to control the photophysical and photochemical properties of various molecules, such as fluorescence enhancement and intramolecular excimer/exciplex formation [1–10]. Over the past two decades, we have investigated the solvent, pH, and CD dependences of the photophysical properties of various molecules [1–10] in both the ground and excited states.

Since different organic molecules exhibit remarkable behavior depending on pH and microenvironmental conditions, it is worthwhile to study some substituted phenols under diverse conditions. In this paper, we investigated the behavior of 2-phenylenediamine (2PDA) in the presence of α -CD and β -CD, which are widely used as model systems for studying cyclodextrin inclusion complexation. The present work focuses on: (i) To analyse absorption and fluorescence spectral shifts of 2PDA in α -CD, β -CD and solvents of different polarities (ii) proton-transfer behavior of 2PDA in aqueous, α -CD, and β -CD media; (iii) the structures and geometries of the inclusion complexes using PM3 molecular modeling; and (iv) the doping effect of 2PDA:CD on Ag nanomaterial is analyzed through DSC, FTIR, ¹H NMR, and SEM techniques [1–10].

MATERIALS AND METHODS

Preparation of CD Solution

The concentration of the stock solution of 2PDA was 2×10^{-2} mol/dm³. Aliquots of the stock solution (0.1 or 0.2 mL) were transferred into 10 mL volumetric flasks. Varying concentrations of α -CD or β -CD solutions ($0.2, 0.4, 0.6, 0.8$, and 1.0×10^{-2} mol/dm³) were added. The mixed solutions were diluted to the mark with triply distilled water and shaken thoroughly. The final concentration of 2PDA in all flasks was 4×10^{-4} mol/dm³. All experiments were carried out at room temperature (298 K).

Preparation of Ag:2PDA:CD Nanomaterials

A 0.01 M solution of silver nitrate was prepared in 50 mL of deionized water and warmed at 50–60 °C for 30 minutes. Then, 1–2 mL of 1% trisodium citrate solution (1 g dissolved in 100 mL of deionized water) was added with vigorous stirring. The appearance of a pale yellow color confirmed the formation of silver nanoparticles [11–16].

Cyclodextrin (1 mmol) was dissolved in 40 mL of distilled water, and 2PDA (1 mmol) dissolved in 10 mL of ethanol was slowly added to the CD solution. The mixture was stirred at 50 °C for 2 hours using a magnetic stirrer. Subsequently, the silver nanoparticle solution was added and stirred for an additional 2 hours. The resulting dilute solution was gently warmed at 40–50 °C until its volume was reduced by approximately 50%. The solution was then refrigerated overnight at 5 °C.

The precipitated Ag–2PDA–CD nanomaterials were collected by filtration and washed several times with small amounts of ethanol and water to remove uncomplexed 2PDA, silver, and CD, respectively. The product was dried under vacuum at room temperature and stored in an airtight container. The resulting powder samples were used for further characterization and analysis [11–16].

RESULT AND DISCUSSION

Effect of α -CD and β -CD on 2PDA

Table 1, Fig. 1 and Fig. 2 represent the absorption and emission spectral maxima of 2-phenylenediamine (2PDA, 2×10^{-4} M) in pH~3, and pH~7 solutions in different α -CD and β -CD concentrations. To compare the inclusion behavior of the neutral and monocationic species of the 2PDA molecule with CD, the complexation behaviour was studied in pH~3, and pH~7 solutions.

The absorption and emission maxima of 2PDA in absence of CD at pH~3, and pH~7 aqueous solutions appear in the following wavelength: (pH~3: $\lambda_{\text{abs}} \sim 274, 252$ nm, $\lambda_{\text{flu}} \sim 306, 360$ nm; pH~7: $\lambda_{\text{abs}} \sim 288, 252$ nm, $\lambda_{\text{flu}} \sim 306, 363$ nm).

The above results indicate that, the neutral species present in pH~7, whereas the blue shifted absorption maxima indicate monocation exists in pH~3. In pH~7, the emission maximum at 360 nm resembles the spectra observed in non-aqueous solvents and thus can be assigned to the molecular form of 2PDA.

In both pH solutions, with increasing the CD concentrations, the absorption maxima of the 2PDA decreased at the same wavelength in the α -CD, whereas increased in the β -CD. In the excited state, upon increasing the CD concentrations, the absorption maxima of the 2PDA decreased at the same wavelength in the α -CD/pH~3, while increased in α -CD/pH~7 solution.

In the absence and presence of α -CD and β -CD solutions, the absorption and emission maxima and spectral shape of 2PDA in pH~3 is different from pH~7 buffer solutions. In α -CD medium, no noteworthy absorption

spectral change detected in $pH \sim 3$ and $pH \sim 7$, while in β -CD, the absorption maximum is red shifted (288 to 294 nm). In both the pH s, absorbance of 2PDA increased in α -CD solution.

The effect of β -CD on the absorption and emission spectra of 2PDA is more pronounced than the corresponding effect on the α -CD. With on increasing the α -CD concentrations, the emission intensities decreased at the same wavelength in $pH \sim 3$, while increased in $pH \sim 7$. At $pH \sim 3$, α -CD with 2PDA, single emission maximum noted at 360 nm, but at $pH \sim 7$, dual emission noted at 295, 360 nm.

Table 1 Absorption and fluorescence maxima of 2-phenylenediamine (2PDA) with different α -CD and β -CD concentrations.

Concentration of CD ($\times 10^{-3}$ M)	pH – 3 λ_{abs} (nm)	log ϵ	λ_{flu} (nm)	τ	pH – 7 λ_{abs} (nm)	log ϵ	λ_{flu} (nm)	τ
2PDA only (without CD)	274, 252	3.98	360	0.049	288, 252	4.05	306, 363	0.031
0.2 α -CD	274, 252	3.96	360	0.059	288, 252	4.02	306, 363	0.060
1.0 α -CD	274, 252	3.87	360	0.062	288, 252	3.93	306, 363	0.064
0.2 β -CD	274, 258, 239	4.04	295, 360	0.065	294, 252	4.04	296, 362	0.077
1.0 β -CD	274, 258, 239	4.07	295, 360	0.073	294, 252	4.06	296, 362	0.086
Excitation wavelength (nm)	270				270			
K (1:1) $\times 10^5$ M $^{-1}$ α -CD	81.1		289		174		355	
ΔG (kcal mol $^{-1}$) α -CD	-11.08		-14.29		-13.00		-14.80	
K (1:1) $\times 10^5$ M $^{-1}$ β -CD	122		337		129		315	
ΔG (kcal mol $^{-1}$) β -CD	-12.11		-14.67		-12.26		-14.50	

In the ground state, β -CD variation of absorbance noticed in the 2PDA molecule suggest excimer is formed. Upon increasing the β -CD concentrations, the emission intensities decreased in both the pH s. Further, in β -CD, dual emission is noticed both in the $pH \sim 3$ and $pH \sim 7$. Further, in β -CD, at $pH \sim 3$, the emission intensity decreased at the shorter wavelength (SW, normal emission) whereas increased in the longer wavelength (LW). However, at $pH \sim 7$, both the SW and the LW emission intensities decreased at the same wavelength. An addition of both α -CD and β -CD, the change in the absorbance and emission intensity has been recognized to the enhanced dissolution of the 2PDA molecule through the encapsulation of the guest in to the CD cavity [17-30] indicating the formation of 2PDA:CD inclusion complex. At higher CD concentrations, the absorption and emission maxima and the spectral shape of 2PDA in both the pH solutions are different suggests different inclusion complex may be formed. In addition, no noteworthy changes were watched in the absorbance of these solutions when recorded after 12 hrs. In both the pH solutions, the presence of isosbestic point in the absorption spectra suggests 1:1 inclusion complex is formed but the orientation of the guest molecule in to the CD cavity may be different [17-30]. The binding constant (K) values were obtained from the slope and the intercept of the Benesi-Hildebrand plots. A plot of $1/A - A_0$ versus $1/[CD]^2$ and $1/I - I_0$ versus $1/[CD]^2$ (both absorption and emission) gives an upward curve, while a plot of $1/A - A_0$ versus $1/[CD]$ and $1/I - I_0$ versus $1/[CD]$ reveals a linear relationship. This analysis reflects the formation of 1:1 inclusion complex between 2PDA:CD. The thermodynamic parameter ΔG for the formation of the guest molecule to CD is given in Table

1. As can be seen from the Tables, ΔG is negative which suggests that the inclusion is an exothermic process and proceeded simultaneously at 303K.

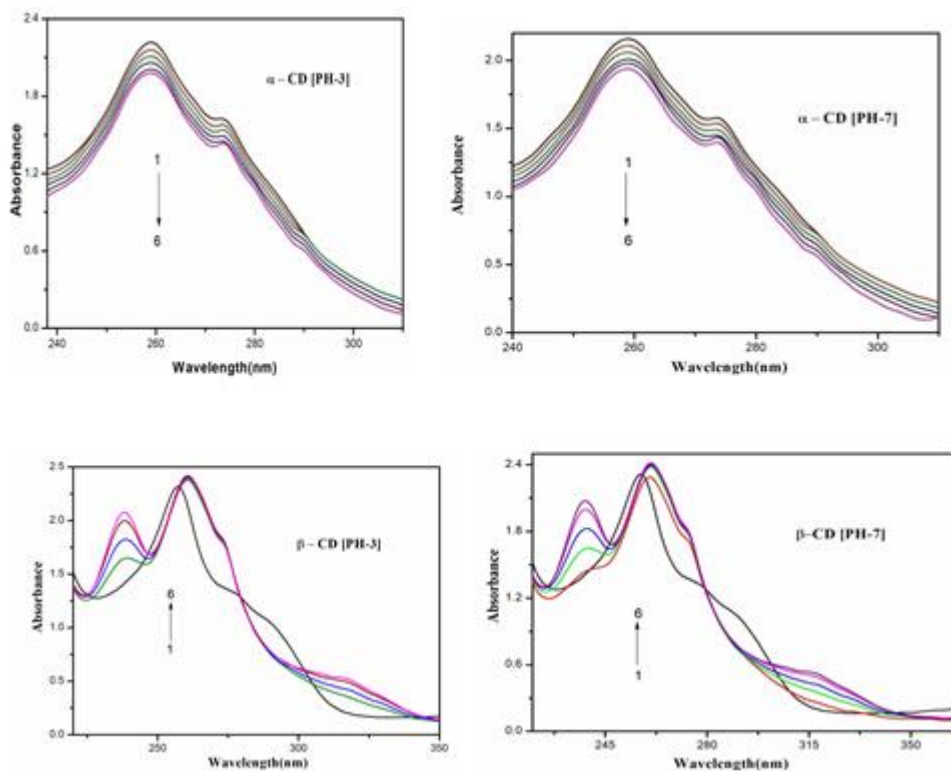


Fig. 1 Absorbance spectra of 2PDA in different α -CD and β -CD concentrations (M): 0, (2) 0.002, (3) 0.004, (4) 0.006, (5) 0.008, (6) 0.01.

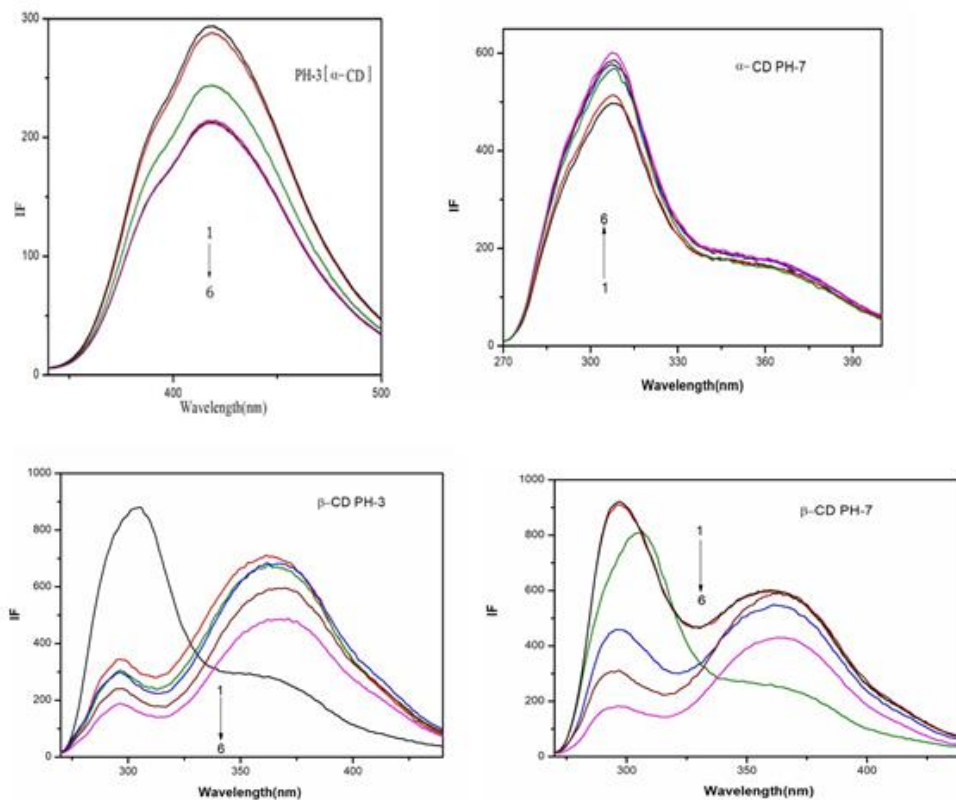


Fig. 2 Fluorescence spectra of 2PDA in different α -CD and β -CD concentrations (M): 0, (2) 0.002, (3) 0.004, (4) 0.006, (5) 0.008, (6) 0.01.

Excimer Emission

2PDA/ α -CD exhibits single emission at pH~3, while dual emission noted in other solutions; i.e., 2PDA/ α -CD at pH~7, 2PDA/ β -CD at pH~3, and pH~7. The dual emission typical of excimer can be formed were explained in our earlier articles. Compared to higher CD concentrations, the excimer emission is very weak in the lower CD concentrations. This is because the variations of polarity, viscosity and CD cavity size may play a more important role in the change in the excimer behaviour of the 2PDA molecule [16-30].

To check the dual emission in 2PDA with CD, we also studied the solvent provoked changes in the absorption and emission spectra for this molecule in selected solvents. The spectral maxima of the 2PDA in the selected solvents are given below (cyclohexane: λ_{abs} ~290, 234 nm, λ_{flu} ~330; acetonitrile: λ_{abs} ~294, 239 nm, λ_{flu} ~350 nm; methanol: λ_{abs} ~293, 237 nm, λ_{flu} ~354; water: λ_{abs} ~287, 232 nm, λ_{flu} ~355). The results show that, in the solvents the absorption and emission maximum of 2PDA is similar to 3-aminophenol (3AP) [31] (cyclohexane: λ_{abs} ~286, 236 nm, λ_{flu} ~314; acetonitrile: λ_{abs} ~288, 239 nm, λ_{flu} ~324; methanol: λ_{abs} ~283, 232 nm, λ_{flu} ~320; water: λ_{abs} ~281, 232 nm, λ_{flu} ~336). 2PDA gave a single broad emission spectrum in all the solvents. The absence of longer wavelength emission in 2PDA indicates that excimer or Intramolecular Charge Transfer (ICT) or exciplex is not formed in all the solvents. On comparison to aniline (cyclohexane: λ_{abs} ~283, 235 nm, λ_{flu} ~320 nm; acetonitrile: λ_{abs} ~286, 238 nm, λ_{flu} ~329; methanol: λ_{abs} ~284, 232 nm, λ_{flu} ~334; water: λ_{abs} ~278, 230 nm, λ_{flu} ~335 nm) the absorption maxima of 2PDA are close in all the solvents.

In all the solvents 2PDA gives single emission maximum while dual luminescence in CD. The dual emission is explained as follows: Among the two maxima one occurs in shorter wavelength region (300 nm, SW) and the other in longer wavelength region (360 nm, LW). As the β -CD concentration increased, the emission maxima of both SW and LW bands shift to red, and the shift being greater for the SW band. The reason for the dual emissions is already discussed in our earlier articles [16-30].

Excited Singlet State Lifetimes

To examine the CD provoked changes in the fluorescence spectra of 2PDA the emission decay of the normal and ICT emissions in aqueous α -CD and β -CD solutions were analysed (Table 1). In 2PDA, biexponential decay was noticed in the water and α -CD/pH-3, whereas in α -CD/pH-7 β -CD with pH-3 and pH-7 solution, a triexponential decay noticed. The decay behavior of 2PDA indicates the existence of the two different emitting species that compete with the conformational relaxation times required for the excimer. The decay time of the low intensity was similar to that of excimer emission. This indicated that equilibrium between the locally excited (LE) state and the excimer was achieved in water in a rather short period. However, in the presence of β -CD, the equilibrium between the LE and excimer states was changed due to the formation of the CD inclusion complexes. The decay time of the LE state slightly increased from water to α -CD and β -CD.

The decay time of the excimer emission was observed to be very short in addition of β -CD influenced by the inclusion process. This behavior indicates the competing process between the excimer and the normal emission. Upon addition of α -CD, the excimer emission decay profile was weak triexponential decay without significant enhancement of the lifetime. On contrary, upon addition of β -CD same decay appeared in the excimer emission suggesting the formation of different 2PDA: α -CD and 2PDA: β -CD inclusion complexes. The lifetimes of the guest: host inclusion complexes were higher than that of the isolated guest molecules. The increase in the lifetime value with increase in CD concentration is due to the encapsulation of the 2PDA in the CD cavity. The life time of the 2PDA increased in the following order: water < α -CD < β -CD. This order indicates that β -CD:2PDA inclusion complex has more stable than α -CD inclusion complexes. This reflects that the ICT dynamics of the 2PDA: α -CD inclusion complex is quite different from that of the 2PDA: β -CD complexes.

Molecular Modeling

The ground state geometries of 2PDA and CDs were optimized using PM3 method (Fig. 3). HOMO, LUMO, thermodynamic parameters (energy, enthalpy, entropy and free energy), dipole moment, zero point vibrational

energy and Mulliken charge of the 2PDA, α -CD and β -CD and inclusion complexes are summarized in Table 2. Both CDs heights are same (7.8 Å) and the interior cavity size of α -CD is 4.7- 5.3 Å while β -CD is 6.0 - 6.5 Å and the exterior cavity size of α -CD is 8.8 Å and β -CD is 10.8 Å. The interior and exterior cavity size of α -CD is lower than that of β -CD. In 2PDA, the vertical and horizontal bond distance between H-H is 5.46 Å and 4.97 Å respectively (Fig. 3). In 2PDA, both vertical and horizontal bond length of the 2PDA is lower than β -CD cavity size. The vertical bond length of 2PDA is lower but horizontal bond length is higher than α -CD cavity size. Since the horizontal bond length of 2PDA is greater than the dimensions of the α -CD, the guest molecule cannot be fully present inside of the CD cavity. The above results indicate that, 2PDA can form different type of inclusion complex in the α -CD and β -CD. Further, the optimized structures of the inclusion complexes were also confirmed that the guest molecules partially included in the CD cavity.

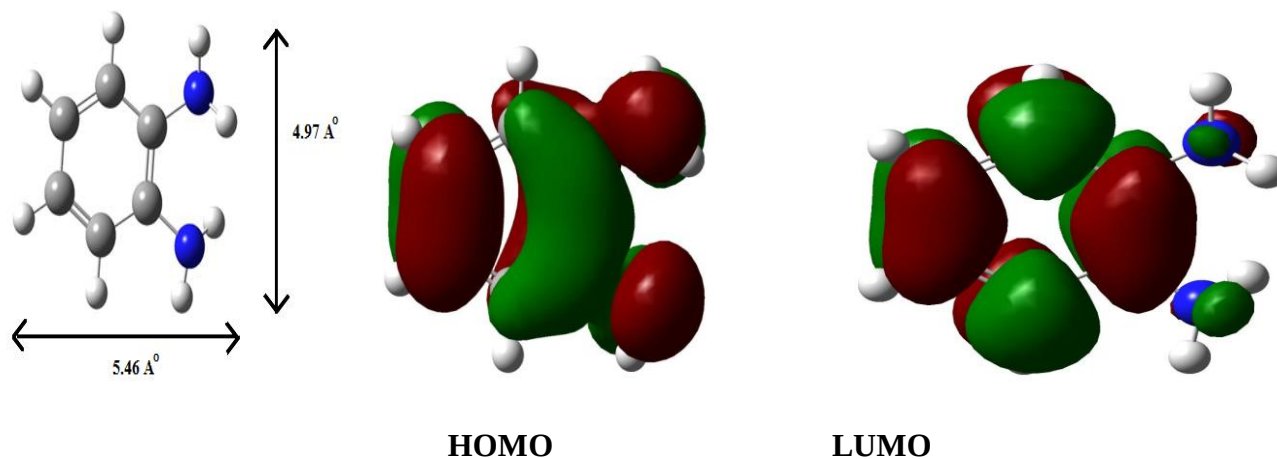


Fig. 3 PM3 optimized structures of (a, 2PDA (b, c) HOMO, LUMO of 2PDA

Table 2. Energetic features, thermodynamic parameters and HOMO-LUMO energy calculations for 2PDA and its inclusion complex by PM3 method.

Properties	2PDA	α -CD	β -CD	2PDA- α -CD A	2PDA- α -CD B	2PDA- β -CD A	2PDA- β -CD B
E_{HOMO} (eV)	-8.16	-10.05	-9.99	-8.08	-8.07	-7.96	-7.96
E_{LUMO} (eV)	0.34	0.14	0.12	0.10	0.15	0.14	0.14
$E_{\text{HOMO}} - E_{\text{LUMO}}$ (eV)	-8.50	-10.19	-10.11	-8.18	-8.23	-8.10	-8.10
μ (eV)	-3.91	-4.95	-4.93	-3.99	-3.96	-3.91	-3.91
χ (eV)	3.91	4.95	4.93	3.99	3.96	3.91	3.91
η (eV)	4.25	5.09	5.05	4.09	4.11	4.05	4.05
S (eV)	2.12	2.54	2.52	2.04	2.05	2.02	2.02
ω (eV)	3.59	4.81	4.81	3.89	3.81	3.77	3.77
Dipole (D)	0.55	9.92	10.52	9.20	8.47	9.34	8.73
E^*	22.12	-1353.95	-1577.74	-1339.58	-1339.4	-1564.01	-1563.12
ΔE^*				-7.75	-7.59	-8.40	-7.50
G^*	57.76	510.13	606.37	585.92	587.17	679.86	681.61
ΔG^*				18.02	19.26	15.72	17.47
H^*	82.42	599.76	704.03	680.69	679.93	784.50	783.98
ΔH^*				-1.49	-2.25	-1.95	-2.47

S**	82.72	300.59	327.58	317.85	311.13	350.97	343.35
ΔS^{**}				-65.46	-72.18	-59.33	-66.95

* kcal mol⁻¹ ** kcal/mol-Kelvin

The thermodynamic parameter ΔG for the formation of the guest molecule to both CDs are negative which suggests that the inclusion proceeded simultaneously at 303 K. The experimental results indicate that the inclusion reactions were exothermic process. HOMO, LUMO, energy, free energy, enthalpy, entropy, dipole moment and zero point vibration energy of the CD:2PDA is appreciably changed than the isolated guest molecule indicates inclusion complex is formed. The polarity of the CD changed after the guest entered in to the CD cavity. The negative energy, enthalpy and Gibbs free energy changes suggested that the inclusion processes were energetically and enthalpically favourable in nature. The binding energy (ΔE) of both the inclusion complexes are higher than that of isolated 2PDA molecule suggesting that stability of complexes are more. The negative ΔH values indicated that the inclusion formation of 2PDA with CD is an exothermic and enthalpy driven. The small negative ΔS value is due to enhancement of disorder in the system. Moreover hydrophobic interactions, which are long range interactions, can be important in the CD complex formation. Comparison of ΔH and ΔS confirm that enthalpy change is higher and entropy changes are lower for the complexation. Therefore, the inclusion complex of 2PDA with CD is more enthalpy stabilized.

Inclusion Complex Nanomaterial Studies

Scanning Electron Microscopy

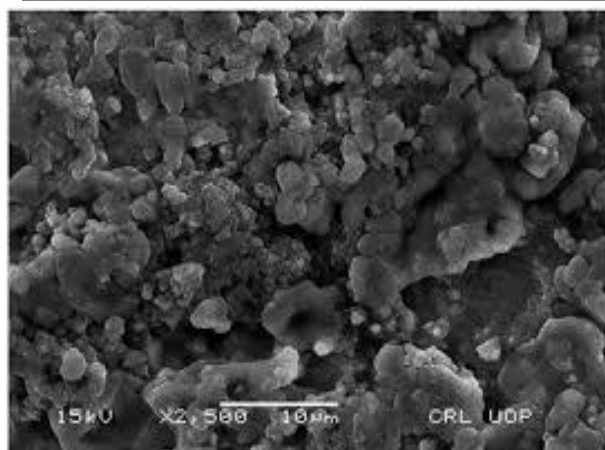
The powdered form of Ag nano, 2PDA, Ag:2PDA: α -CD and Ag:2PDA: β -CD nanomaterials were investigated by SEM (Fig.4). These pictures clearly show, Ag nano present in ball shape, 3PDA in ball shape, Ag:3PDA: α -CD and Ag:3PDA: β -CD are in rock shape. SEM EDEX data confirm 34.2% carbon, 44.2% oxygen and 21.6% nano Ag present in the nanomaterials. The different structure of pure nano Ag, 2PDA and the inclusion complex supports the formation of the Ag-2PDA-CD nano materials. Modification of these morphologies can be taken as a proof for the formation of a new Ag:2PDA:CD inclusion complex nanomaterials.

Differential Scanning Colorimeter

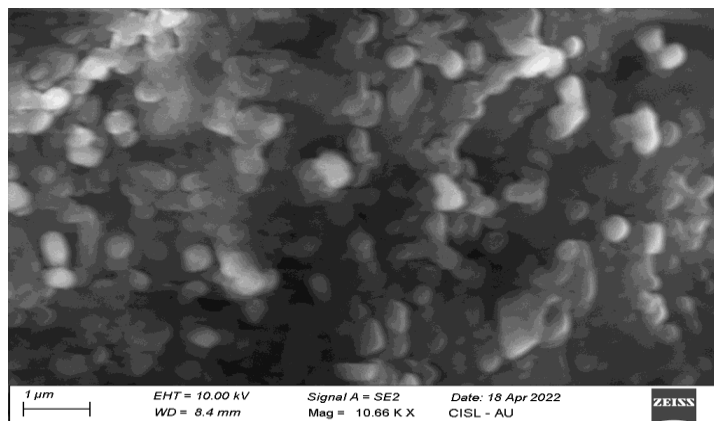
The DSC profiles of α -CD, β -CD, 2PDA and the Ag:2PDA: α -CD and Ag:2PDA: β -CD nanomaterials are analysed. The DSC curves of α -CD show three endothermic peak at 79.2 °C, 109.1 °C and 137.5 °C and β -CD shows a broad endothermic peak at 128.6 °C and, these endothermic peaks are attributed to crystal water loss from CDs. The melting point of 2PDA shows a sharp peak at 104 °C. A broader endothermic effect was recorded for α -CD, β -CD and respective inclusion complexes as a consequence of water loss from the CDs. The DSC thermogram of inclusion complexes did not show peaks corresponding to pure 2PDA and CD, instead new peaks appeared at 255 °C and 276 °C for 2PDA: α -CD and 2PDA: β -CD respectively.

Infrared Spectral Studies

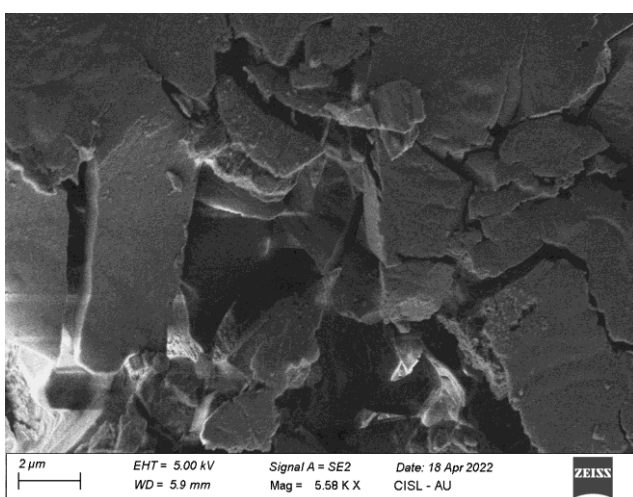
FTIR spectra shows the Ag:2PDA: α -CD and Ag:2PDA: β -CD nanomaterials in comparison to those for pure α -CD, β -CD and 2PDA. In 2PDA, the -NH stretching frequency appears at 3383, 3363 cm⁻¹ and -C-H stretching peak appears at 3026 cm⁻¹. The aromatic C-H, and C-C stretching frequency appears at 2671 cm⁻¹ and 849 cm⁻¹ respectively. In the Ag:2PDA:CD nanomaterials, the aromatic C-H and C=C stretching frequency appears at 2890 cm⁻¹ and 1626 cm⁻¹ respectively. The NH₂ was moved in the nanomaterials to 3280 cm⁻¹. Ag nano was moved in the nanomaterials to 575 cm⁻¹. In Ag:2PDA:CD most of the frequency was not appeared and significant decrease in intensity was noted suggest that the 2PDA molecule interact with silver nano and CD.



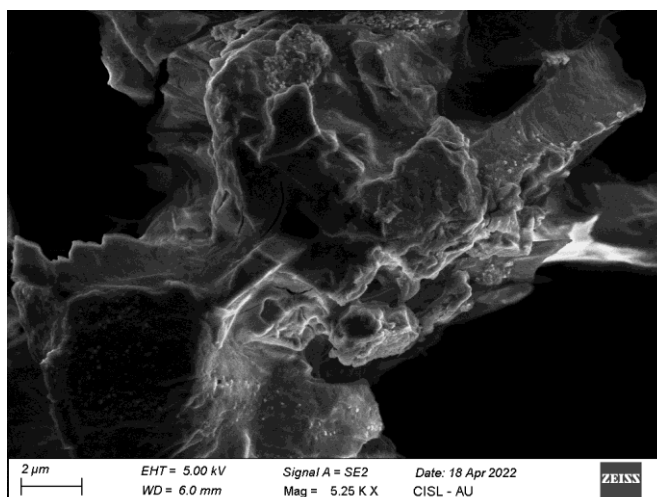
a) Ag nano



b) 2PDA



c) Ag-2PDA- α -CD



d) Ag-2PDA- β -CD

Fig. 4 SEM photographs of (a) 2PDA, (b) nano Ag:2PDA: α -CD, (b) nano Ag:2PDA: β -CD.

H NMR Spectral Studies

^1H -NMR spectra of 2PDA and the inclusion complexes are performed at 25 °C in $\text{DMSO}-d_6$. Generally, the chemical shift values of the 2PDA protons tend to show appreciable changes if the guest molecules are included in the CDs cavities.

Both amino protons appear 4.35, the ortho proton (2^{nd} and 5^{th} position) appear at 6.32 (doublet) and the 3^{rd} and 4^{th} protons appear at 6.504 (doublet). The chemical shift value of 2PDA protons are shifts to up field in the both CD complexes. These results indicate that all the protons of 2PDA are interacting with CD cavity protons.

X-RD Spectral Studies

XRD analysis confirmed the formation of nanomaterials. Based on JCPDS data, the mineral name (3C) and face-centered cubic (FCC) structure were identified. The standard FCC structure corresponds to JCPDS card number 87-0717, with hkl values at 111, 200, 220, and 311.

Ag nanoparticles showed four distinct peaks at $2\theta = 38.11^\circ$, 44.30° , 64.45° , and 77.40° . Three peaks were observed for 2PDA at $2\theta = 17.24^\circ$, 19.66° , 28.86° . Ag/2PDA/ β -CD showed nine peaks at $2\theta = 12.65^\circ$, 19.84° , 15.25° , 26.82° , 38.13° , 40.14° , 55.13° , 64.18° , and 77.84° . The XRD patterns of Ag/2PDA/ β -CD exhibited

distinct diffraction features, confirming the formation of new nanomaterials. The appearance of additional peaks and variations in intensities further support the formation of novel nanomaterials.

CONCLUSION

Absorption and emission spectral maxima of 2-phenylenediamine (2PDA) in different α -CD and β -CD concentrations with $pH \sim 3$, and $pH \sim 7$ solutions were investigated. In the absence and presence of α -CD and β -CD solutions, the absorption and emission maxima and spectral shape of 2PDA in $pH \sim 3$ is different from $pH \sim 7$ buffer solutions.

2PDA gives single emission maximum in α -CD but dual emission in β -CD. 2PDA gave a single broad emission spectrum in all the solvents while the dual emission in the CD solutions indicates that excimer is formed in the CD solutions. The geometrical restriction of the α -CD cavity would restrict the free rotation of the amino groups. This lifetime values indicates that β -CD:2PDA inclusion complex has more stable than α -CD inclusion complexes.

The thermodynamic parameter values are significantly changed than the isolated guest molecule indicates inclusion complex is formed. The powdered form of Ag nano, 2PDA, Ag:2PDA: α -CD and Ag:2PDA: β -CD inclusion complexes were investigated by SEM, DSC, FTIR and 1H NMR. SEM pictures, EDEX data confirm 34.2% carbon, 44.2% oxygen and 21.6% nano Ag present in the nanomaterials. DSC and FTIR values of the Ag:2PDA:CD are different from the isolated 2PDA, CD Ag nanomaterials.

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