Synthesis of Silver– 4,4'-diaminodiphenyl –Cyclodextrin Nanomaterials and pH Dependent Characteristics of 4,4'-diaminodiphenyl – Cyclodextrin Inclusion Complexes

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ABSTRACT

The spectral characteristics of 4,4'-diaminodiphenyl (benzidine, DADP) 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. Ag:DADP:CD nanomaterials were synthesized and characterized by SEM, DSC, FTIR, ^{1}H NMR and XRD techniques. Both pH conditions, DADP exhibited distinct absorption and emission shifts upon complexation with α-CD and β-CD. DADP showed a single broad emission band in all solvents, and CD solutions indicates the presence of twisted intramolecular charge transfer (TICT) in the DADP molecule. The fluorescence lifetimes of the inclusion complexes were greater than that of free DADP. In the DADP molecule, both the vertical and horizontal bond lengths between the amino and hydroxy groups are smaller than the β-CD cavity size. SEM–EDX data confirmed the presence of 5.5% silver in the nanomaterials.

Keywords : 4,4'-diaminodiphenyl, cyclodextrin, silver nano, pH effects, TICT

INTRODUCTION

The most important property of CD is their ability to admit a variety of appropriately sized guest molecules into the cavity with formation of inclusion complexes [1-10]. Although a large number of systems are capable of forming inclusion complexes [33-37] with CDs, the changes in the photophysical properties on encapsulation are in many cases too small to provide any meaningful and reliable information on the micro-heterogeneity of the CD or the location of the probe. This necessitates further studies of the complexation processes involving probes whose photophysical properties are sensitive to any small changes in its environment. In the present study we are examining the effect of pH in α-CD and β-CD and inclusion by both CDs on 4,4'-diaminodiphenyl (DABP). In this context, the present work analyzes: (i) the absorption and fluorescence spectral shifts and the first excited singlet-state lifetime of DADP in α-CD, β-CD, solvents of different polarities, and at various pH values; (ii) the proton transfer reaction of DADP in aqueous, α-CD, and β-CD media; (iii) the structures and geometries of the inclusion complexes by molecular modeling (PM3) methods; and (iv) the doping effect of DADP:CD on silver nanomaterials characterized by DSC, FTIR, ^{1}H NMR, and SEM techniques [1-10].

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MATERIALS AND METHODS

Preparation of CD Solution

The concentration of the stock solution of 4,4'-diaminodiphenyl (DADP) 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 DADP in all flasks was 4×10^{-4} mol/dm³. All experiments were carried out at room temperature (298 K).

Preparation of Ag:DADP: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 DADP (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–DADP–CD nanomaterials were collected by filtration and washed several times with small amounts of ethanol and water to remove uncomplexed DADP, 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].

RESULTS AND DISCUSSION

Absorption and Fluorescence Spectral Results

Absorption and fluorescence maxima of 4,4'-diaminodiphenyl **(**benzidine, DABP) in pH~3 and pH~7 phosphate buffer solutions containing various concentrations of α-CD and β-CD have given in Table 1, Fig. 1 and Fig.2. To evaluate the inclusion behavior of neutral and monocation species of the DABP, studied in *p*H~3 and pH~7 solutions respectively. In absence of CD, the absorption and emission maxima of DABP in the above *p*H solutions appears in the following wavelength: pH~3: λ_{abs}~248 nm, λ_{flu}~420 nm; pH~7: λ_{abs}~282 nm, λ_{flu}~393, nm; The above results indicate that, monocation exists in pH~3 and neutral present in pH~7 respectively (Table 1). In pH~7, the absorption (282 nm) and emission maximum (393 nm) be similar to the spectra observed in non-aqueous solvents hence it can be assigned to the molecular form of DABP (cyclohexane: λ_{abs}~278 nm, λ_{flu}~337 nm; acetonitrile: λ_{abs} ~285 nm, λ_{flu}~380 nm; methanol: λ_{abs}~281 nm, λ_{flu}~380 nm; water: λ_{abs}~280 nm, λ_{flu}~393 nm).

The solvents results shown that, the absorption maximum of DABP is red shifted from cyclohexane to acetonitrile but blue shifted in water while the emission maxima is red shifted from cyclohexane to water. DABP gave a single broad emission in all the solvents. The absence of longer wavelength emission maxima in polar solvents indicates that TICT is not formed in all the solvents. Table 1 reveals that no significant shift noticed between water and solvents in the absorption and emission spectra of DABP. Further, the acidic pH~3, does not affect the appearance of the spectrum except with blue shift. The blue shift in pH~3 suggests protonation takes place in the amino group (monocation formed). The above results consistent with the characteristic of amino and hydroxyl groups [17-30].

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Table 1 Absorption and fluorescence maxima of DABP with different α-CD and β-CD concentrations.

Concentration of CD ($\times10^{-3}$ M)	pH – 3 λabs (nm)	log ε	λflu (nm)	IF	pH – 7 λabs (nm)	log ε	λflu (nm)	IF
DABP only (without CD)	248	4.42	420	0.25	282	4.33	393	0.25
0.2 M α-CD	248	4.12	420	0.32	282	4.31	393	0.35
1.0 M α-CD	248	4.21	420	0.41	282	4.22	393	0.44
0.2 M β-CD	248	4.23	390	0.34	281	4.17	395	0.36
1.0 M β-CD	248	4.05	390	0.43	281	4.10	395	0.48
Excitation wavelength (nm)	260				260			
K (1:1) $\times10^5$ M^{-1} α-CD	316		640		165		304	
ΔG (kcal mol^{-1}) α-CD	-14.50		-16.28		-12.87		-14.44	
K (1:1) $\times10^5$ M^{-1} β-CD	205		612		184		387	
ΔG (kcal mol^{-1}) β-CD	-13.41		-16.18		-13.14		-15.04	

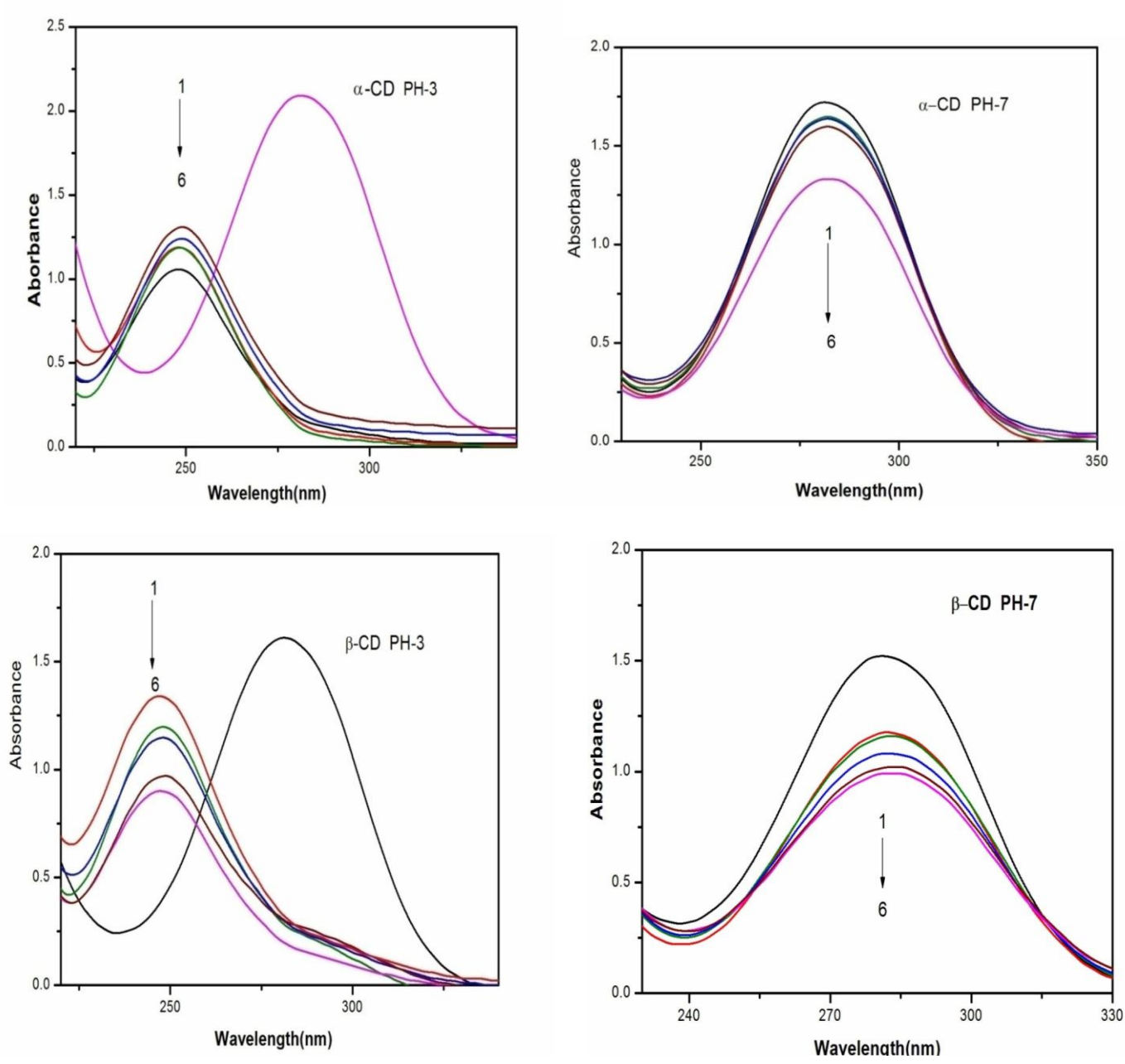

Fig. 1 Absorption spectra of DABP in different α-CD and β-CD concentrations (M): 0, (2) 0.002, (3) 0.004, (4) 0.006, (5) 0.008, (6) 0.01.

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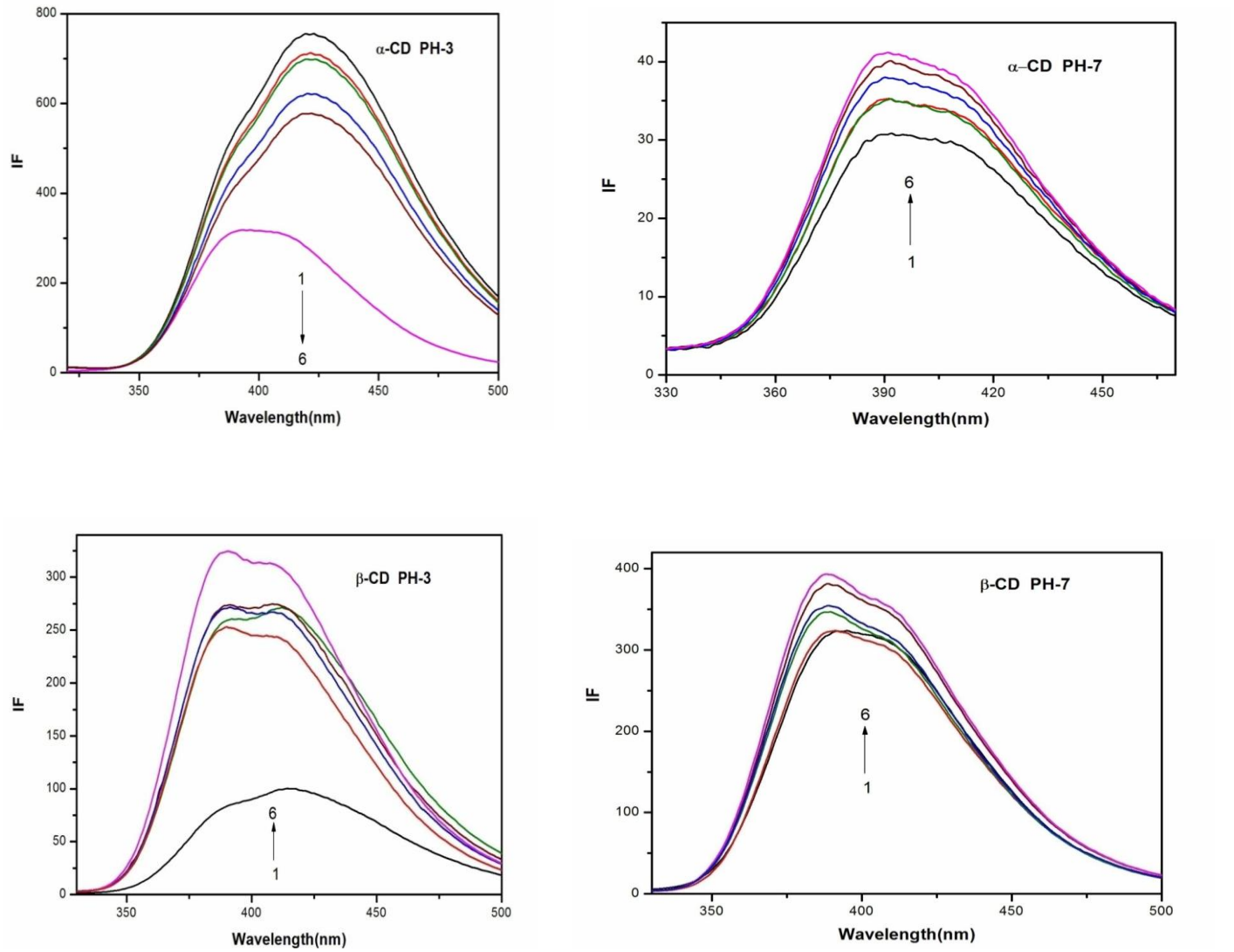

Fig. 2 Fluorescence spectra of DABP in different α-CD and β-CD concentrations (M): 0, (2) 0.002, (3) 0.004, (4) 0.006, (5) 0.008, (6) 0.01.

In the ground state, with an increasing the α-CD and β-CD concentrations, the absorbance of the DADP is decreased/increased at the same wavelength in both pH~3 and pH~7. In the excited state, upon increasing the α-CD concentrations, the emission intensity decreased at the same wavelength in the pH~3 while increased in the pH~7. Further, in β-CD, the emission intensity increased in the pH~3 and pH~7 solutions. Interestingly, a broad emission is observed in all the pHs. In addition, no noteworthy changes were detected in the absorbance of these solutions when recorded after 12 hrs. A similar change observed in the absorbance and emission intensities and the spectral maxima are owing to the encapsulation of DABP molecule into the α-CD and β-CD cavities [17-30].

The effect of α-CD and β-CD on the excited state of DABP is more prominent than the effect on the ground state. The enrichment of the fluorescence intensity in pH~3 is stronger than pH~7. In both CDs in the pHs, similar absorption and fluorescence spectral shifts observed establish that DABP molecule form same type of the inclusion complexes. The binding constant (K) values were obtained from the Benesi-Hildebrand plot of $1/A\text{-}A_0$ *vs* $1/[CD]^2$ and $1/I\text{-}I_0$ *vs* $1/[CD]^2$ (both absorption and fluorescence) gives a linear relationship**.** However, a plot of $1/A\text{-}A_0$ *vs* 1/[CD] and $1/I\text{-}I_0$ *vs* 1/[CD] reveals an upward curve. This analysis reflects the formation of 2:1 inclusion complex between 2DABP:CD. The negative ΔG values (Table 1) suggests that the inclusion proceeded simultaneously at 303K and that the inclusion reaction is an exothermic process. The broad band does appear in the pH solutions suggest monomer may be present. Even though the fluorescence intensity for the longer wavelength (420 nm) was extremely low at all the pHs, broad fluorescence was seen in both the aqueous and CD solutions. The presence of broad longer wavelength emission (390 - 420 nm) intensity indicates TICT present in DADP molecule.

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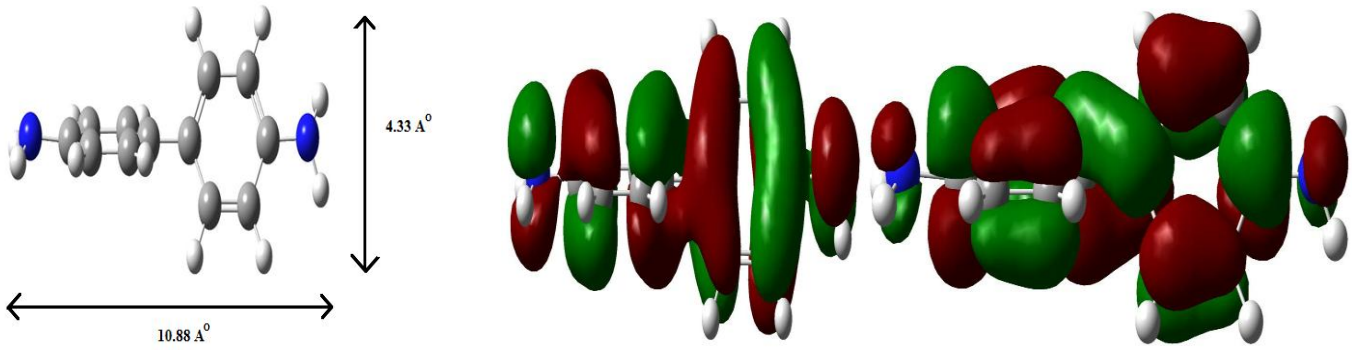
Excited Singlet State Lifetimes

To examine the CD induced changes in the fluorescence spectra of DABP the emission decays of the normal (390 nm) was measured in water and 0.01 *M* α-CD and β-CD (Table 1). The decay of DABP is dependent on the CD. Biexponential decay was observed in aqueous, α-CD and β-CD solutions. The lifetimes of the guest: host inclusion complexes were higher than that of the isolated guest molecules. The life time of the DABP increased in the following order: water < α-CD < β-CD. This order indicates that β-CD:DABP inclusion complex has more stable than α-CD inclusion complexes. The increase in the life time value with increase in CD concentration is due to the encapsulation of the DABP in the CD cavity. The life time values depend on the type of CD and the nature of the process with regard to short-lived species. This may be due to the vibrational restriction of DABP in the excited state.

Molecular Modeling

The ground state geometries of DABP, α-CD and β-CD were optimized using PM3 method. The PM3 level optimized structures of the isolated guest, host and the inclusion complexes are shown in Fig. 3. HOMO, LUMO, thermodynamic parameters (energy, enthalpy, entropy and free energy), dipole moment, zero point vibrational energy and Mulliken charge of the DABP, α-CD and β-CD and inclusion complexes are summarized in Table 2. Both CDs heights are same (7.8 Å), the interior cavity size of α-CD is 4.7- 5.3 Å and 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 DABP, the vertical and horizontal bond distance between NH_2 – NH_2 is 4.33 Å and 10.88 Å respectively (Fig. 3). In DABP, the vertical and horizontal bond length between the amino and groups are lower than α-CD and β-CD cavity size whereas the horizontal bond length higher than α-CD and β-CD cavity size. Considering the shape and dimensions of DABP can be entrapped in the α-CD and β-CD cavity. Further, the optimized structures of the inclusion complexes were also confirmed that the guest molecules included in the CD cavity.



HOMO **LUMO**

Fig. 3 PM3 optimized structures of (a, b) DABP (c, d) HOMO, LUMO of DABP

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

Properties	**DABP**	**DABP-α-CD**	**DABPβ-CD**	**DABP-α-CD A**	**DABP-α-CD B**	**DABP-β-CD A**	**DABP-β-CD B**
E_{HOMO} (eV)	-8.04	-10.05	-9.99	-8.00	-8.22	-7.83	-7.84
E_{LUMO} (eV)	0.27	0.14	0.12	0.11	0.17	-0.02	0.17
$E_{HOMO} - E_{LUMO}$ (eV)	-8.32	-10.19	-10.11	-8.12	-8.39	-7.80	-8.01
μ (eV)	-3.88	-4.95	-4.93	-3.94	-4.02	-3.92	-3.83
χ (eV)	3.88	4.95	4.93	3.94	4.02	3.92	3.83
η (eV)	4.15	5.09	5.05	4.05	4.19	3.90	4.00
S (eV)	2.07	2.54	2.52	2.02	2.09	1.95	2.00
ω (eV)	3.63	4.81	4.81	3.83	3.86	3.94	3.67

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Dipole (D)	2.41	9.92	10.52	11.74	7.88	8.90	10.20
E^*	42.44	-1353.95	-1577.74	-1322.47	-1322.47	-1546.91	-1544.69
ΔE^*				-10.95	-10.95	-11.61	-9.39
G^*	103.98	510.13	606.37	633.04	632.28	726.47	728.78
ΔG^*				18.91	18.16	16.11	18.42
H^*	132.18	599.76	704.03	729.74	729.89	834.32	833.77
ΔH^*				-2.19	-2.05	-1.89	-2.44
S^{**}	94.56	300.59	327.58	324.32	327.36	361.72	352.14
ΔS^{**}				-70.83	-67.79	-60.42	-70.00
ZPE	0	0	0	0	0	0	0

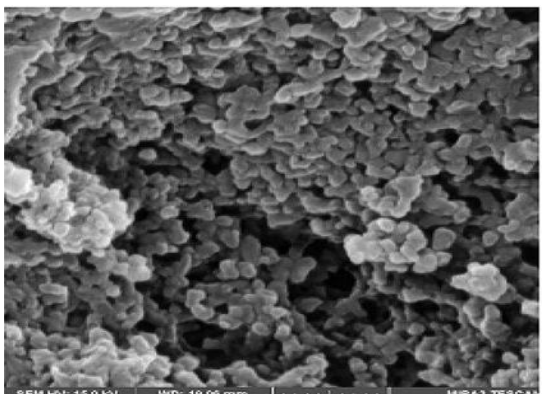
*kcal/mol; **kcal/mol-Kelvin; ZPE = Zero point vibration energy

The negative energy, enthalpy and Gibbs free energy changes suggested that the inclusion processes were energetically and enthalpically favourable in nature. The negative ΔH values indicated that the inclusion formation of DABP with CD is an exothermic and enthalpy driven. The small negative ΔS value is due to enhancement of disorder in the system.

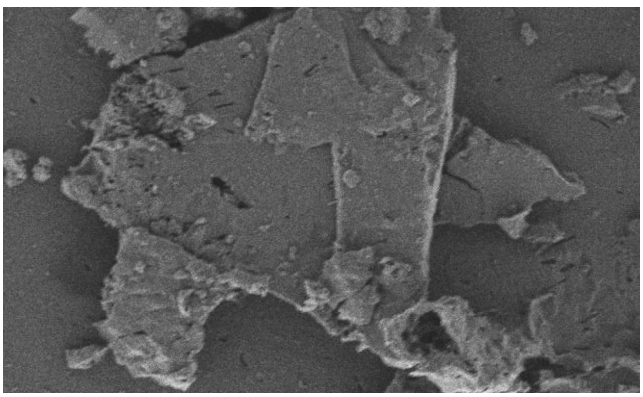
Inclusion Complex Nanomaterials Studies

Scanning Electron Microscopy

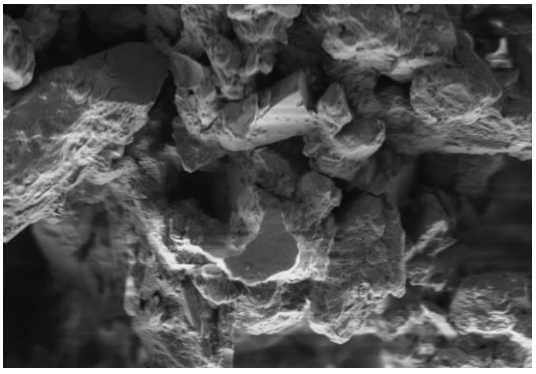
The powdered form of Ag nano, DABP and nano Ag:DABP:α-CD and nano Ag:DABP:β-CD nanomaterials were investigated by SEM (Fig. 4). These pictures clearly show, Ag nano present in ball shape, DADP in sheet shape, Ag:DADP:α-CD and Ag:DADP:β-CD are in rock shape. SEM EDEX data confirm 74.44% carbon, and 21.64 % nano Ag present 5.5 % in the nanomaterials. The different structure of pure nano Ag, DABP and the inclusion complex supports the formation of the Ag-DABP-CD nanomaterials. Modification of these morphologies can be taken as a proof for the formation of a new Ag nano.



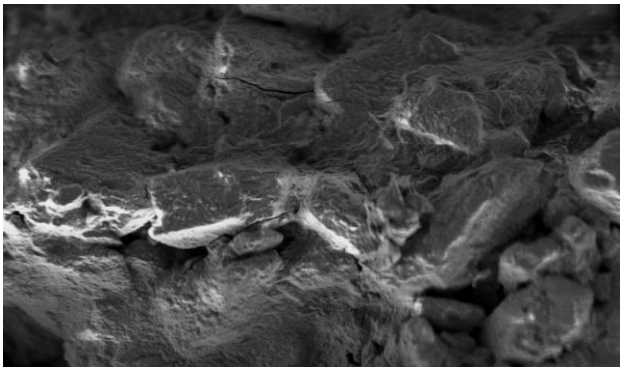
a) Nano Silver



b) DABP



c) Ag -DABP-α-CD



d) Ag-DABP-β-CD

Fig. 4 SEM images for a) Ag. nano, b) DABP, c) Ag. :DABP:α-CD and d) AgDABP:β-CD.

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Differential Scanning Colorimeter

The DSC curves of α-CD shown three endothermic peaks at 79.2 ºC, 109.1 ºC and 137.5 °C and β-CD shows a broad endothermic peak at 128.6 ºC and, these endothermic peaks are attributed to crystal water loss from CDs. The melting point of DABP shows a sharp peak at 128 ºC respectively. A broader endothermic effect was recorded for α-CD, β-CD and respective inclusion complexes as a consequence of water loss from the CDs. DSC thermogram of Cu-DABP-CD complexes did not show peaks corresponding to pure DABP and CD, instead new peaks appeared at 195 ºC and 214 ºC for Cu-DABP-α-CD and Cu-DABP-β-CD respectively.

Infrared spectral studies

In FTIR, the N-H, and C-H stretching frequencies of the isolated DABP molecule appears at 3381, 2718 cm^{-1} respectively. The NH_2 bond deformation and aromatic ring C=C stretching vibration appears at 1653 and 1521 cm^{-1}. The aromatic C-C stretching frequency of DABP appeared at 849, 1173 cm^{-1}, and C-N stretching frequencies appeared at 1250 cm^{-1}.

In the nanomaterials, the NH_2 peak appears at 3264 cm^{-1} and the aromatic C=C and C-O stretching frequencies appeared at 1615 and 1578 cm^{-1} respectively. The aromatic ring deformation appears at C-N stretching frequencies appeared at 1019 cm^{-1}. A substantial decrease in intensity was noted in the nano Ag:DABP:CD complexes suggest that the DABP molecule strongly interact with nano Silver.

Proton Magnetic Resonance Spectral Studies

In order to examine the structures of nano silver, DADP and the CD complexes were investigated by proton magnetic resonance spectra. ^{1}H-NMR spectra of DADP and the inclusion complexes are performed at 25 °C in DMSO-d_6. Generally, the chemical shift values of the guest protons tend to show appreciable changes if the guest molecules are included in the CDs cavities. The DADP chemical shifts values are given below: Aromatic protons (ortho to amine) appear at 6.6–6.8 ppm (doublet, 4H), aromatic protons (meta to amine) seem at 7.3–7.4 ppm (doublet, 4H) and amine protons often visible between 4.5–5.5 ppm. All the protons shift to up field/ down field in the Ag:DADP:CD nanomaterials indicate that all the protons of DADP are interacting with CD cavity protons.

***X*-RD Spectral Studies**

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 2θ = 38.11°, 44.30°, 64.45°, and 77.40°. Three peaks were observed for DADP at 2θ° = 12.6, 15.8, 17.8, 19.8, 29.6, 38.2, 46.4 and 48.3°. Ag/DADP/β-CD showed nine peaks at 2θ° = 14.8, 19.0, 20.4, 22.2, 27.8, 30.4, 34.2, 36.2, 40.1, 46.2, 48.2, 45.7, 48.3, and 77.8°. 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 4,4-diaminodiphenyl (DABP) in different α-CD and β-CD concentrations with *p*H~3, *p*H~7 and silver nano were analysed. Compared to pH~3, the emission intensities are very week in pH~7 medium. In α-CD and β-CD and the pHs, similar absorption and fluorescence spectral shapes observed establish that DABP molecule form similar type of inclusion complexes. The life time of the DABP increased in the following order: water < α-CD < β-CD indicates that DABP:β-CD has more stable than DADP:α-CD inclusion complex. The formation of inclusion complex is further supported by using PM3 calculations. SEM picture clearly shows the morphological difference between silver nano, DADP and the Ag nano. DSC, FTIR, NMR and XRD values of Cu-DABP-CD nanomaterials peaks are different from pure DABP and CD suggest new nanomaterials are formed.

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