Synthesis of Copper:4, 4-Diamino 3, 3-Dimethyl Biphenyl:Cyclodextrin Nanomaterials and pH Dependence of DADMB:CD Inclusion Complexes

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DOI: https://doi.org/10.51584/IJRIAS.2026.11030014

Received: 09 March 2026; Accepted: 14 March 2026; Published: 27 March 2026

ABSTRACT

The spectral characteristics of 4, 4-diamino 3, 3-dimethyl biphenyl (DADMB) with various solvents, α-cyclodextrin (α-CD) and β-cyclodextrin (β-CD) at pH ~3, pH~7, were investigated using UV–visible, fluorescence, time-resolved fluorescence measurements, and PM3 computational methods. The DADMB–CD inclusion complexes and Cu:DADMB:CD nanomaterials were synthesized and characterized by DSC, SEM, FTIR, ^{1}H NMR and XRD techniques. DADMB form 1:2 inclusion complex with CD. In the DADMB molecule, the horizontal bond lengths between the amino groups are higher than the CD cavity size suggest DADMB not fully encapsulate in the CD cavity. The powdered form of Cu nano, DADMB, Cu:DADMB:α-CD and Cu:DADMB:β-CD nanomaterials were investigated by SEM, DSC, FTIR, XRD and ^{1}H NMR. SEM picture clearly shows the morphological difference between copper nano, DADMB and the Cu:DADMB:β-CD. The chemical shift value of DADMB protons are shifts to up field and down field and the peak intensities are very low in the nano copper with CD nanomaterials indicate that all the protons of DADMB are interacting with Cu nano and CD cavity protons.

Keywords: 4, 4-diamino 3, 3-dimethyl biphenyl, cyclodextrin, copper nano, inclusion complex, nanomaterials

INTRODUCTION

Although a large number of systems are capable of forming inclusion complexes [1-10] 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-diamino 3, 3-dimethyl biphenyl (DADMB, also known as-tolidine).

In this work, the present work analyzes: (i) the absorption and fluorescence spectral shifts and the first excited singlet-state lifetime of DADMB in α-CD, β-CD, solvents of different polarities, and at various pH values; (ii) the proton transfer reaction of DADMB 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 DADMB:CD on silver nanomaterials characterized by DSC, FTIR, ^{1}H NMR, and SEM techniques [1-10].

MATERIALS AND METHODS

Preparation of CD Solution

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

Synthesis of Cu:DADMB:CD nanomaterials

A 100 ml solution of $CuSO_4$ (1×10^{-3} mol/dm³) in a round-bottom flask was reduced by the dropwise addition of 1% sodium borohydride while stirring vigorously on a hot plate with a magnetic stirrer. As the reaction proceeded, the solution color changed from pale blue to reddish brown. Subsequently, 5 ml of 1% trisodium citrate was added dropwise as a stabilizing agent.

Separately, α-CD or β-CD (1 mmol) was dissolved in 40 ml of distilled water, and DADMB (1 mmol) dissolved in 10 ml ethanol was added slowly to the CD solution. The mixture was stirred at 50 °C for 2 hours. The prepared copper nanoparticle solution was then added to this mixture and stirred for an additional 2 hours at 40–50 °C. The resulting solution was freeze-dried using a mini-lyophilizer at –80 °C to obtain a powdered product. The Cu:DADMB:CD nanomaterial was washed with small amounts of ethanol and water to remove unreacted DADMB, copper, and CD. The purified precipitate was dried under vacuum at room temperature and stored in an airtight container. The resulting Cu:DADMB:CD powder samples were used for further characterization [11-16].

RESULTS AND DISCUSSION

Effect of α-CD and β-CD on 4, 4- diamino 3, 3- dimethyl biphenyl

Absorption and fluorescence maxima of 4, 4- diamino 3, 3- dimethyl biphenyl (DADMB) 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 DADMB was studied in *p*H~3 and pH~7 solutions respectively. In absence of CD, the absorption and emission maxima of DADMB in the *p*H~3 and pH~7 solutions appear in the following wavelength: pH~3: λ_{abs}~250 nm, λ_{flu}~420 nm; pH~7: λ_{abs}~280 nm, λ_{flu}~385 nm; The above results indicate that, the monocation exists in pH~3 and neutral present in pH~7 respectively. In pH~7, the absorption and emission maximum be similar to the spectra observed in non-aqueous solvents, hence it can be assigned to the molecular form of DADMB. Table 1 values reveals that absorption spectrum of DADMB in water (pH~3) is blue shifted than pH~7 solution. The blue shift in pH~3 suggests protonation takes place in the amino group (monocation formed). However, the emission spectrum is red shifted in the pH~3 solutions than pH~7. The unusual red shift in the pH~3 solution is already explained in our earlier publications; i.e., due to formation of the solute- solvent exciplex red shift is formed in the pH~3 solutions than pH~7 [17-30]. Both in the ground and excited states, with an increasing the α-CD and β-CD concentrations with pH~3 and pH~7 the absorption and emission intensities are decreased at the same wavelength in the DADMB.

The changes observed in the absorbance and emission intensities and the spectral maxima are owing to the encapsulation of DADMB molecule into the α-CD and β-CD cavities. In addition, no noteworthy changes were detected in the absorbance of these solutions when recorded after 12 hrs. In both CDs with pH solutions, the absence of isosbestic point in the absorption spectra with similar spectral shift suggests same type of the inclusion complex formed between DADMB and CDs. The effect of α-CD and β-CD on the excited state of

DADMB is more prominent than the effect on the ground state. The enrichment of the fluorescence intensity in pH~7 is stronger than pH~3.

Concentration of CD x10^{-3} M	**pH - 3**			**pH - 7**		
	λ_{abs}	log ε	λ_{flu}	λ_{abs}	log ε	λ_{flu}
DADMB only (without CD)	250	4.41	420	282	4.34	385
0.2 M α-CD	250	4.25	420	281	4.38	385
1.0 M α-CD	250	4.06	420	281	4.24	385
0.2 M β-CD	250	4.12	420	281	4.14	385
1.0 M β-CD	248	3.96	420	281	4.09	385
Excitation wavelength (nm)		270			270	
K (1:1) x10^5 M^{-1} α-CD	116		-199	252		575
ΔG (kcal mol^{-1}) α-CD	-9.26		-17.3	-9.2		-44.0
K (1:1) x10^5 M^{-1} β-CD	297		227	278		454
ΔG (kcal mol^{-1}) β-CD	-14.3		-7.87	-9.0		-15.7

Table 1. Absorption and fluorescence maxima of 4,4'-Diamino 3,3'-dimethylbiphenyl (DADMB) with different α-CD concentrations.

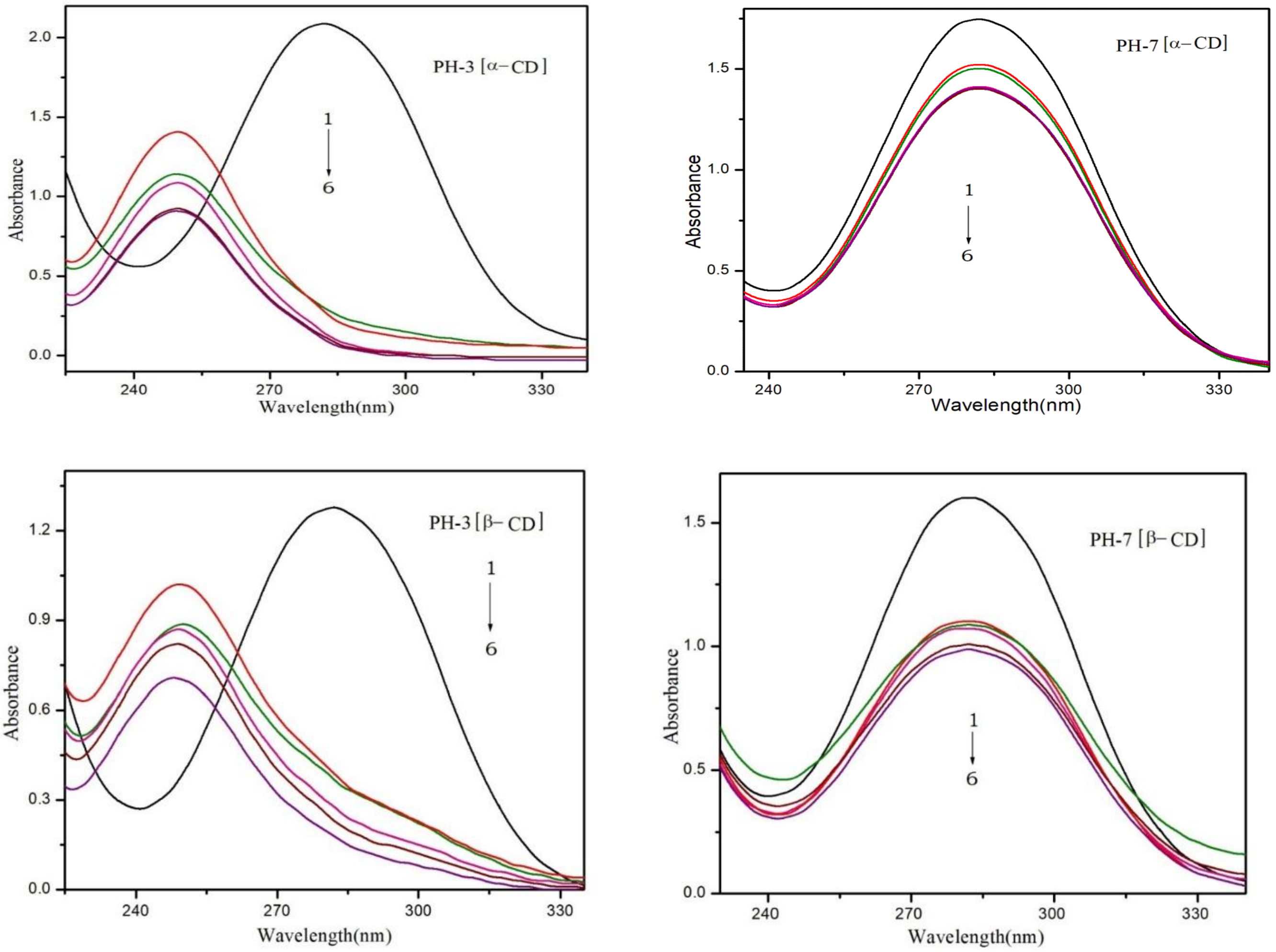


Fig. 1 Absorbance spectra of DADMB in different α-CD and β-CD concentrations (M): 0, (2) 0.002, (3) 0.004, (4) 0.006, (5) 0.008, (6) 0.01.

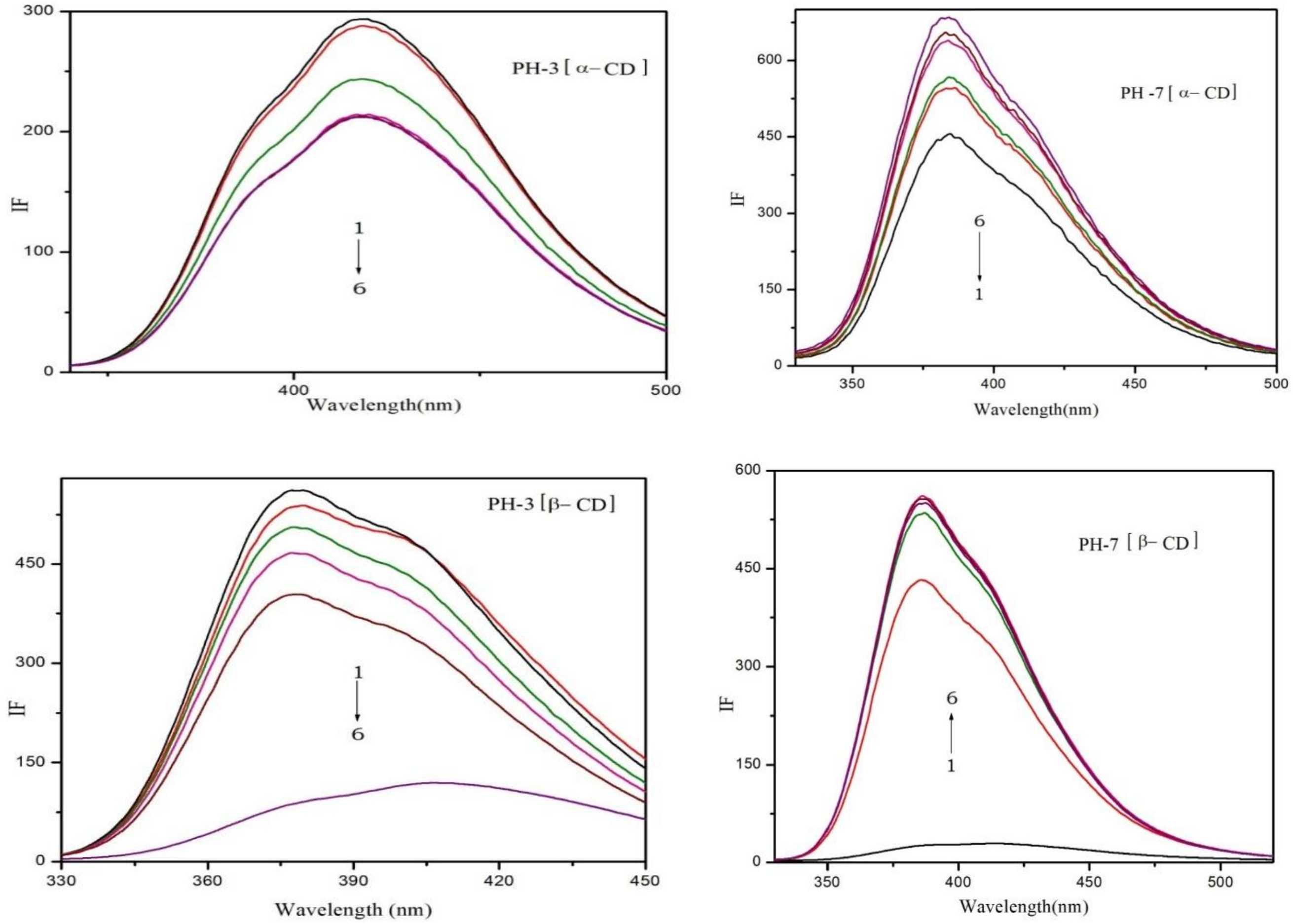


Fig. 2 Fluorescence spectra of DADMB in different α-CD and β-CD concentrations (M): (1) 0, (2) 0.002, (3) 0.004, (4) 0.006, (5) 0.008, (6) 0.01.

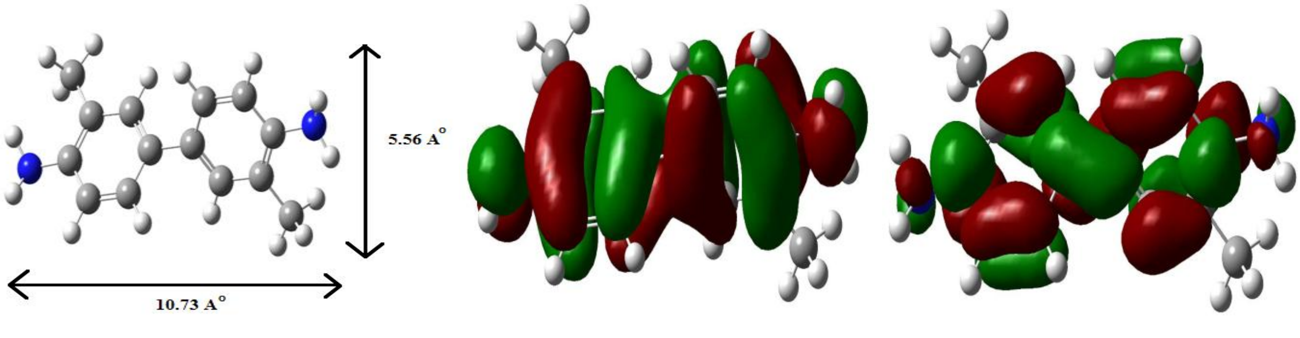
The binding constant (K) values were obtained from the slope and the intercept of the Benesi-Hildebrand plots. A plot of $1/\ A\text{-}A_0$ *versus* $1/[CD]^2$ and $1/\ I\text{-}I_0$ *versus* $1/[CD]^2$ (both absorption and fluorescence) gives a linear relationship**.** However, a plot of $1/A\text{-}A_0$ *versus* $1/[CD]$ and $1/I\text{-}I_0$ *versus* $1/[CD]$ reveals an upward curve. This analysis reflects the formation of 1:2 inclusion complex between DADMB:CD. The negative ΔG values (Table 1) suggests that the inclusion proceeded simultaneously at 303K and that the inclusion reaction of DADMB with CD is an exothermic process.

The presence of broad emission in CD solutions suggests that DADMB may form 1:2 inclusion complex with two CDs (i.e., one DADMB molecules were entrapped in two CD cavity) [14,15]. With an increase in the CD concentration, the emission intensities were decreased, accompanied by a broadening of the band. The decrease in the fluorescence intensity may be 1:2 inclusion complex formed by the 1:2 DADMB:2CD inclusion complex. This is already explained in our earlier publications [17-30].

To prove the complexation between the DADMB and α-CD/ β-CD, we also studied the solvent provoked changes in the absorption and fluorescence spectra for this molecule in selected solvents. The results shows that, in the solvents the absorption maximum of DADMB is red shifted from cyclohexane to acetonitrile but blue shifted in alcohol and water while the emission maxima is red shifted from cyclohexane to water (cyclohexane: λ_{abs}~305, 236 nm, λ_{flu}~337 nm; acetonitrile: λ_{abs} ~308, 240 nm, λ_{flu}~350 nm; methanol: λ_{abs}~300, 236 nm, λ_{flu}~362 nm; water: λ_{abs}~295, 232 nm, λ_{flu}~385 nm). DADMB gave a single broad fluorescence spectrum in the solvents. The absence of longer wavelength emission maxima in polar solvents indicates that ICT or exciplex is not formed in all the solvents. Further, when compared to phenol and aniline the absorption maxima of DADMB are red shifted in all the solvents (phenol in water: λ_{abs}~272-278 nm, λ_{flu}~330; aniline in water: λ_{abs}~278 nm, λ_{flu}~335) indicates delocalization between the amino and hydroxy groups are large.

Molecular Modeling

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 DADMB, α-CD and β-CD and inclusion complexes are summarized in Table 2. 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 DADMB, the vertical and horizontal bond distance between $NH_2 – NH_2$ is 10.73 Å and 5.56 Å respectively (Fig. 3). In DADMB, the horizontal bond length between the both amino groups are higher than α-CD and β-CD cavity size. Considering the shape and dimensions of DADMB 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 [31-39].



HOMO **LUMO**

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

Table 2 Energetic features, thermodynamic parameters calculations for DADMB and its inclusion complexes by PM3 method.

Properties	DADMB	α-CD	β-CD	DADMB -α-CD A	DADMB -α-CD B	DADMB -β-CD A	DADMB -β-CD B
E_{HOMO} (eV)	-8.13	-10.37	-10.35	-8.44	-8.51	-8.12	-8.22
E_{LUMO} (eV)	-0.002	1.26	1.22	-0.39	-0.47	0.01	0.15
$E_{HOMO} – E_{LUMO}$ (eV)	-8.12	-11.63	-11.57	-8.04	-8.03	-8.14	-8.38
μ (eV)	-4.06	-4.55	-4.56	-4.41	-4.49	-4.05	-4.03
χ (eV)	4.06	4.55	4.56	4.41	4.49	4.05	4.03
η (eV)	4.06	5.81	5.78	4.02	4.02	4.06	4.18
S (eV)	2.03	2.90	2.89	2.01	2.01	2.03	2.09
ω (eV)	2.03	1.78	1.80	2.42	2.50	2.02	1.94
Dipole (D)	2.00	11.34	12.28	10.52	10.08	13.27	10.70
E^*	27.39	-1246.41	-1456.33	-1226.96	-1227.54	-1441.39	-1438.73
ΔE^*				-7.94	-8.52	-12.45	-9.78
G^*	173.37	-675.16	-694.78	-457.96	-452.55	-509.40	-510.25
ΔG^*				43.82	49.23	12.00	11.15
H^*	210.62	-569.72	-600.00	-339.81	-334.35	-398.60	-396.57
ΔH^*				19.28	24.74	-9.21	-7.18
S^{**}	124.93	353.64	317.88	396.29	396.44	371.63	381.30
ΔS^{**}				-82.28	-82.13	-71.17	-61.50
ZPE	0	0	0	0	0	0	0

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

The thermodynamic parameters of the DADMB:CD is significantly 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 negative ΔH values indicated that the inclusion formation of DADMB 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

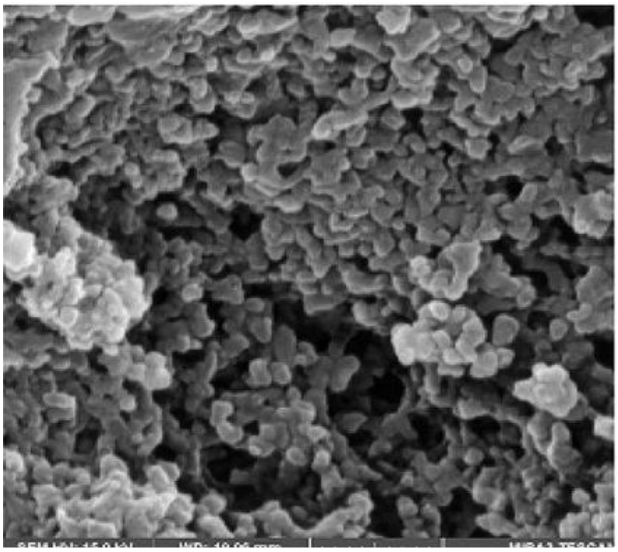
Scanning Electron Microscope

The powdered form of copper nano, DADMB and Cu:DADMB:α-CD and Cu:DADMB:β-CD nanomaterials were investigated by SEM (Fig. 4). **Nano Copper (a):** The image typically reveals small, discrete nanoparticles. These often exhibit a high surface-area-to-volume ratio, appearing as semi-spherical or irregular clusters depending on the synthesis method.

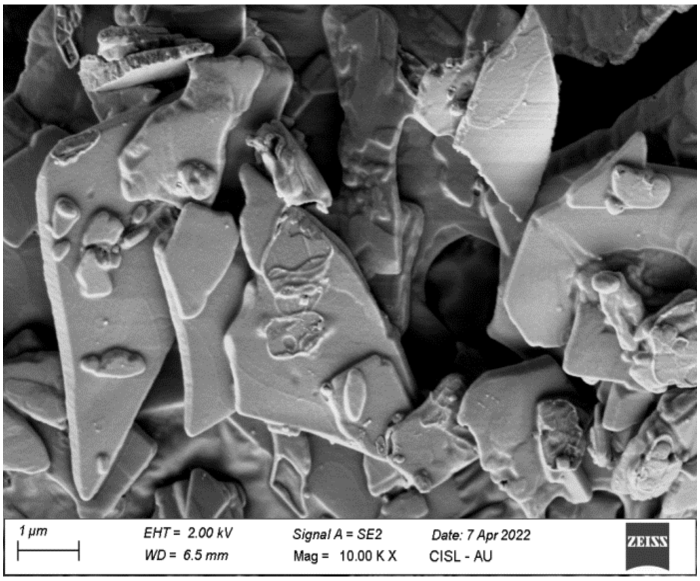
DADMB (b): The DADMB, generally shows a distinct crystalline or layered structure. In SEM, this often manifests as elongated micro-rods or plate-like shapes.

Cu:DADMB:-CD (c): Upon the addition of α-CD, the morphology shifts. The interaction between the copper nanoparticles, the organic linker (DADMB), and the macrocyclic α-CD often leads to more structured, integrated aggregates, potentially forming micro-sheets or network-like structures.

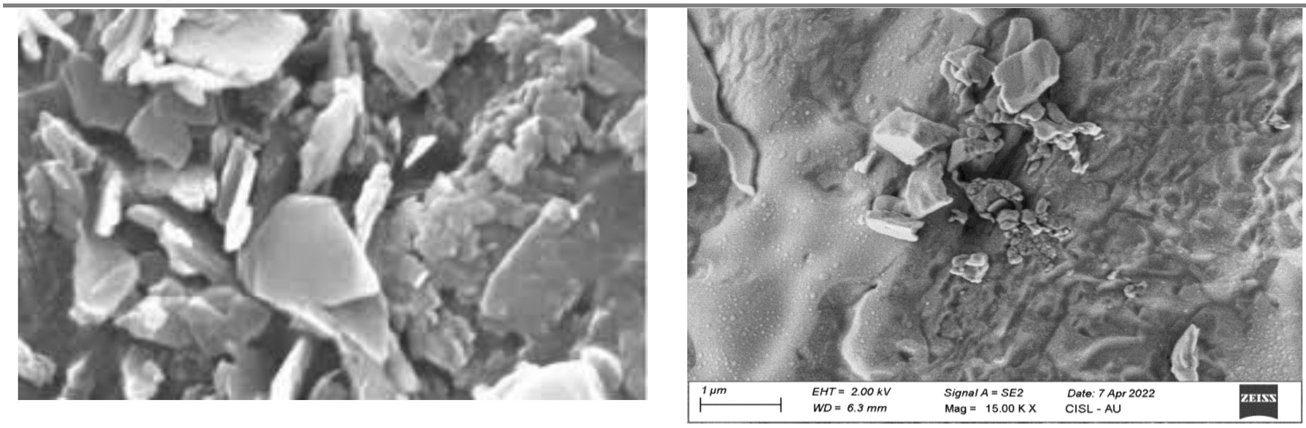
Cu:DADMB:β-CD (d): The β-CD complex typically displays a different texture compared to the α-CD version. Because β-CD has a larger hydrophobic cavity, it may facilitate the formation of larger, more defined crystalline blocks or "flower-like" hierarchical structures as the components self-assemble. SEM EDEX data confirm 46.8% carbon, 49.0% oxygen and 2.9% nano Cu present in the nanomaterials. The transition from image (a) and (b) to (c) and (d) confirms the formation of a composite. The encapsulation or stabilization by cyclodextrins significantly alters the particle size and surface topography, which is crucial for applications in catalysis or sensing.



a) Nano Copper



b) DADMB



c) Cu -DADMB-α-CD **d) Cu-DADMB-β-CD**

Fig 4 SEM images for a) Cu nano, b) DADMB, c) Cu:DADMB:α-CD and d) Cu:DADMB:β-CD

Differential Scanning Colorimeter

DSC analysis reveals distinct thermal transitions for the α-CD and β-CD the guest molecule DADMB, and their respective copper-based inclusion complexes: Cyclodextrin Dehydration: The native CDs exhibit endothermic peaks due to the loss of crystalline water. For α-CD, this occurs in three stages at 79.2 ºC, 109.1 ºC and 137.5 °C, while β-CD displays a single broad endotherm at 128.6 ºC. The melting point of DADMB is characterized by a sharp endothermic peak at 131 ºC, indicating high crystallinity. The characteristic melting peaks of pure DADMB and the dehydration peaks of the CDs disappear, replaced by new thermal signatures for Cu-DADMB-α-CD and Cu-DADMB-β-CD at 195 ºC and 214 ºC respectively. This shift to higher temperatures suggests enhanced thermal stability and encapsulation of the guest molecule within the cyclodextrin cavity. The DSC thermograms for Cu-DADMB-α-CD and Cu-DADMB-β-CD confirm the formation of new chemical environments.

Infrared spectral studies

In FTIR, the N-H, $-CH_3$ and C-H stretching frequencies of the isolated DADMB molecule appears at 3371, 2982 and 3063 cm^{-1} respectively. The NH_2 bond deformation and aromatic ring C=C stretching vibration appears at 1621 and 1570 cm^{-1}. The aromatic C-C, C-N stretching of 879 cm^{-1} and 1315 cm^{-1} respectively in the DADMB molecule. In the nanomaterials, the NH_2 and $-CH_3$ peaks appears at 3271cm^{-1} and 2923 cm^{-1} and the aromatic C=C stretching frequency appeared at 1621, 1343 cm^{-1} respectively. The aromatic ring deformation appears at 564 cm^{-1}. The C-N stretching frequency appeared at 1315 cm^{-1}. A substantial decrease in intensity was noted in the nano Cu:DADMB:CD complexes suggest that the DADMB molecule strongly interact with nano copper.

Proton Magnetic Resonance Spectral Studies

In order to comprehensively examine the structures of nano copper, DADMB and the CD complexes were investigated by proton magnetic resonance spectra [31-39]. ^{1}H-NMR spectra of DADMB and the inclusion complexes are performed at 25 °C in DMSO-d_6. The NMR spectrum of DADMB reflects a highly symmetrical structure, where both halves of the biphenyl system are equivalent. In a standard solution such as, the spectrum typically displays the following features: The DADMB chemical shifts values are given below: Aromatic protons (ortho to methyl) appear at 7.23 ppm (doublet, 2H), aromatic protons (para to methyl) seem at 7.21 ppm (doublet, 2H), aromatic protons (meta to methyl) seem at 6.68 ppm (doublet, 2H), two equivalent amine group protons (4H) often visible between 3.56 ppm and two equivalent methyl group protons (6H) often visible between 2.19 ppm. A minor shift is observed for the resonance of the protons. The chemical shift value of DADMB protons are shifts to up field in the nano copper with CD nanomaterials. These results indicate that all the protons of DADMB are interacting with copper nano and 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. The Cu nano shows three distinct diffraction peaks at 2θ = 43.58°, 51.64°, and 74.75°. Three peaks were observed for DADMB at 2θ° = 13.6, 16.8, 20.8, 25.8, 32.6, 39.2, 44.4 and 49.3°. Cu/DADMB/β-CD showed nine peaks at 2θ° = 14.8, 18.6, 23.8, 24.8, 38.0, 44.1, 64.3, 77.4 and 113.4°. The XRD patterns of Cu/DADMB/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 DADMB in different α-CD and β-CD concentrations with *p*H~3 and *p*H~7 solutions were analysed using UV-visible, fluorescence, time resolved fluorescence measurements and molecular modeling methods. The absorption and emission spectra of DADMB with pH~ 3 resemble with pH~7 suggest that DADMB molecule form similar types of the inclusion complexes. DADMB form 1:2 inclusion complex with CD. The horizontal bond length of DADMB is greater than the dimensions of the α-CD, the guest molecule cannot be fully present inside of the CD cavity. The powdered form of Cu nano, DADMB, Cu:DADMB:α-CD and Cu:DADMB:β-CD inclusion complexes were investigated by SEM, DSC, FTIR, XRD and ^{1}H NMR. SEM picture clearly shows the morphological difference between copper nano, DADMB and the Cu:DADMB:β-CD. DSC, FTIR, XRD and ^{1}H NMR values of Cu:DADMB:CD different from pure DADMB and CD suggest new nanomaterials formed. The chemical shift value of DADMB protons are shifts to up field and down field and the peak intensities are very low in the nano copper with CD nanomaterials indicate that all the protons of DADMB are interacting with Cu nano and CD cavity protons.

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