CHAPTER 3

SYNTHESIS OF ADSORBENTS

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3.1 Materials and methods

Montmorillonite, MMT – KSF (Catalog No. 69907) was obtained from Sigma Aldrich (USA). Chitosan was obtained from Everest Biotech. Cetylpyridinium chloride, CPC (Catalog No. 588393) and Cetyl trimethylammonium bromide, CTAB (Catalog No. 52365), were obtained from Merck (Germany) and British Drug Houses Ltd. (BDH, England), respectively. Leuco crystal violet (LCV) (Catalog No. 219215) was obtained from Sigma Aldrich Pvt. Ltd. (Germany).

Arsenious oxide (Catalog No. 214987), potassium antimonyl tartarate were obtained from British Drug House (London), whereas vanadium pentoxide was obtained from Merck (Germany). Sodium hydroxide and potassium iodate were obtained from Qualigens (India), hydrochloric acid from RFCL Ltd. and acetic acid was obtained from Merck. Chitosan (DD 78.0%) was obtained from Everest Biotech.

The AR grade chemicals/reagents and double distilled water was used throughout the experiment.

3.2 Methodology for synthesis of modified Montmorillonite, MMT-1

Organoclay (surfactant modified clay), were synthesized by the modification of the reported procedure [001]. About 5·0g of MMT (dried at 80°C) was allowed to stand for
24 hours in 250 ml of double distilled water. To this, 2.0% CTAB solution was gradually added under constant stirring [Figure 3.1].

![Figure 3.1. Schematic representation of synthesis of CTAB modified MMT, MMT-1](image)

After two hours, the clay suspension was recovered by centrifugation and the process was repeated after each washing with double distilled water, for the removal of unreacted surfactant. The residues thus obtained were dried at 353K and labeled as MMT-2.

### 3.3 Methodology for synthesis of modified Montmorillonite, MMT-2

The CPC (surfactant) modified MMT was synthesized using MMT as the host clay. The procedure adopted for the synthesis was same as used for CTAB modified MMT [Chapter 3, Section 3.2].
3.4 Methodology for synthesis of LCV modified Montmorillonite, MMT-3

The Leuco crystal violet (LCV) modified Montmorillonite was synthesized by optimization of the sorption process of LCV on MMT.

200.0mg adsorbent + 100.0mL LCV solution → Centrifugation at 8000rpm → Residue + Filtrate → Dried at 28±2°C → Characterized by XRD, FTIR, TGA, DTA, SEM, TEM → Conc. estimated using UV-VIS

**Figure 3.2. Schematic representation of synthesis of LCV modified MMT, MMT-3**

About 200mg of MMT (dried at 80°C) was continuously agitated with 100ml of 250μl LCV at a predetermined pH using a batch shaker. The solutions from the batch samples were centrifuged [Figure 3.2]. Adsorption experiments were also investigated as a function of pH (1·0-13·0), contact time (01-180 minutes) and initial concentration of LCV solution.

3.5 Methodology for synthesis of Chitosan beads, CHITO-B

Chitosan beads were synthesized by the modification of the reported procedures [002]. 200mg of chitosan flakes was allowed to stand overnight in 100ml 1·0% (v/v) acetic acid solution. The resulting solution obtained was sprayed through a syringe into 0·5M NaOH solution to form hydrogel beads [Figure 3.3].
The obtained chitosan hydrogel beads were allowed to stand in aqueous NaOH for 1 hour for hardening. The beads were finally separated out from the NaOH solution and were washed with double distilled water until the solution became neutral.

### 3.6 Methodology for synthesis of Chitosan Montmorillonite beads, MMT-4

Chitosan Montmorillonite beads were synthesized by the modification of the reported procedures [003]. 200mg of chitosan flakes and 300mg of Montmorillonite was allowed to stand overnight in 13·0ml of 1·0% (v/v) acetic acid. The resulting gel obtained was sprayed into 0·5M NaOH solution to form hydrogel beads through a syringe.

Thus synthesized Chitosan Montmorillonite beads were allowed to stay in NaOH solution for some time. The beads were finally taken out from NaOH solution and were washed with double distilled water until the solution pH became neutral [Figure 3.4].
3.7 Methodology for synthesis of modified Montmorillonite, MMT-5

Chitosan modified MMT was synthesized by modification of reported procedure [004]. Chitosan solution was prepared by addition of corresponding amount of polysaccharide to 25ml of 1% v/v acetic acid solution and stirring for 4hours [Figure 3.5].
Prior to the addition of the chitosan solution to the clay suspension, the pH of the polysaccharide solution was adjusted to 4.9 with NaOH to avoid any structural alteration of MMT.

### 3.8 Characterization of the adsorbents

**X-Ray Diffractometry**

The X-ray diffractograms of Montmorillonite (MMT) and modified MMT have been studied as shown in Figure 3.6. In addition to adsorption onto external surface, intercalation also takes place in phyllosilicates as evident from the diffractograms.

The \(d_{(001)}\) spacing was found to be dependent on the amount of surfactant intercalated. The decrease in 20 value was observed in case of MMT-2 and MMT-1 with respect to MMT. The \(d_{(001)}\) spacing of MMT, MMT-2 and MMT-1 was found to be 12.0 Å, 19.5 Å and 17.6 Å respectively. The increase in \(d_{(001)}\) spacing of MMT-2 and MMT-1 with respect to MMT was found to be 7.5 Å and 5.6 Å, respectively that could be attributed to the movement of surfactant into the intergallery space or in other words to the intercalation of surfactant into the interlayer region of the host clay, MMT.

The \(d_{(001)}\) spacing of MMT-3 was found to be 23.0 Å, indicating an increase of 10.0 Å with respect to MMT [Table 3.1]. On the other hand, an increase of 10.2 Å is observed in the \(d_{(001)}\) spacing of MMT-5 (23.2 Å) with respect to MMT. The X-ray diffractogram as shown in Figure 3.6(a, b) indicates the manner in which LCV and chitosan are intercalated and thus contributes towards increase in gallery space \((d_{(001)}\) spacing\) between MMT layers.

In MMT-3, LCV is represented by the monomeric cationic specie being located in the interlayer space with the aromatic ring parallel to the MMT layers and bound to oxygen planes through \(\pi\)–interactions [004].

LCV is capable of forming an interlayer complex with MMT when adsorbed from aqueous solution as also observed for benzidine [005] and shown in Figure 3.7. LCV is attached strongly to MMT by the electron-donor-acceptor association. The unpaired 2pz electron from nitrogen conjugates with \(\pi\)-electron system of aromatic ring. The water presumably acts as a swelling agent facilitating the entry of LCV into the interlayer space.
Figure 3.6. X-Ray Diffractograms of (a) MMT (b) MMT-1 (c) MMT-2 (d) MMT-3 (e) MMT-5
Table 3.1. The d (001) and 2θ values for various adsorbents

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<thead>
<tr>
<th>S.No.</th>
<th>Adsorbent</th>
<th>2θ value (degrees) Before</th>
<th>2θ value (degrees) After</th>
<th>d (001) value (Å) Before</th>
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Figure 3.7. Schematic representation of interaction of (a) LCV (b) chitosan in MMT

The role of water (solvent) may be represented by Equation (3.1):

$$M - L^+ + H_3O^+ \leftrightarrow M - L^{+2} + H_2O$$  (3.1)

“L” represents the LCV whereas “M” represents surface of MMT.

The introduction of an acetylenic bond into the resonating system of triphenylmethane dye makes the cation moiety coplanar by releasing steric congestion between the ortho hydrogen atoms of phenyl groups [006, 007]. The number of resonating structures formed by the blue color monovalent species is more than that formed by the pale yellow (divalent) species thus accounting for its stability. The maximum adsorption of LCV on MMT was attained at pH 4·0.
The color of the complex formed was observed to be dependent on pH of the system. The LCV interacted MMT at pH 1·0 was observed to be colorless that turned to steel blue at pH 3·0. The MMT-3 was observed to change color to blue cornflower at pH 3·0.

Generally, there are two most important mechanisms of adsorbate adsorption on solids: (i) ion exchange and (ii) hydrophobic interactions [008] whereas two well-distinguished phases of adsorbate adsorption on MMT, first can be ascribed to incomplete monolayer formation. After the monolayer onset of respective adsorbate, complete bilayers are formed. Some adsorption arrangements of CPC on MMT are illustrated in Figure 3.8.

Figure 3.8. Possible arrangements of CPC adsorbed onto MMT (a) Incomplete monolayer (b) Monolayer (c) Bilayer (d) Monolayer intercalated (e) Bilayer intercalated [08]

Figure 3.8a-c shows the CPC adsorption on external MMT surface whereas Figure 3.8d-e simply manifest the intercalation of CPC, as observed from X-ray diffractometry. Similar adsorption mechanism is also followed by other adsorbates taken (CTAB, LCV, CHI).
Monolayer intercalation is depicted by less decrease in 2 theta value as compared to bilayer intercalation and thereby leading to lesser increase in $d_{(001)}$ spacing than bilayer [Table 3.1].

❖ **Surface area analysis**

The surface area of MMT, MMT-1 and MMT-2 as 28700cm$^2$g$^{-1}$, 82000cm$^2$g$^{-1}$ and 98200cm$^2$g$^{-1}$, respectively by Nitrogen BET adsorption method. The surface area of MMT-2 and MMT-1 was found to increase to 53300cm$^2$g$^{-1}$ and 69500cm$^2$g$^{-1}$, respectively, with respect to MMT. The surface area of MMT-3 and MMT-5 was found to be 99800cm$^2$g$^{-1}$ and 99790cm$^2$g$^{-1}$, respectively.

![Figure 3.9. Photographic image: (a) CHITO-B (b, b1) MMT-4](image)

The increase in surface area is possibly due to an increase in the $d_{(001)}$ spacing with respect to MMT. The CHITO-B and MMT-4 were found to be spherical in shape as shown in Figure 3.9 that is expected to provide an increased surface area to the composites synthesized.

❖ **Fourier Transform - Infra Red - Attenuated Total Reflectance Spectrometry**

The FT-IR-ATR spectra of MMT, MMT-1, MMT-2, MMT-3, MMT-4, CHITO-B and MMT-5 are shown in Figure 3.10A and Figure 3.10B and tentative assignments of vibrational bands in Table 3.2.

The MMT is characterized by the bands -OH stretching and bending as well as Si–O stretching and bending. A broad, intense hydroxyl-stretching (OH-stretching) band is
observed at 3440 cm$^{-1}$, while OH-bending vibration band occurs at 1636 cm$^{-1}$. The Si–O stretching vibration band is observed at 1047 cm$^{-1}$, whereas bands in the region 400–600 cm$^{-1}$ (459 cm$^{-1}$ and 529 cm$^{-1}$ respectively) are attributed to Si–O and Al–O bending vibration.

In modified MMT (MMT-2 and MMT-1), the OH-stretching vibration was observed with a sharp peak at 3632 cm$^{-1}$ that accounts for the presence of intermolecular hydrogen bonding. The band at 3440 cm$^{-1}$ was found to be unaffected by the presence of CPC and CTAB and is assigned to the vibration of the Montmorillonite clay (MMT).

The C–H stretching vibrations at 2854 cm$^{-1}$ (sym. –CH$_2$) and 2928 cm$^{-1}$ (asym. –CH$_2$–) in CPC–MMT and MMT-1 respectively were found to be absent in MMT, thereby confirming the presence of the organic moiety onto the clay surface [009].

The strong bending vibrations corresponding to Al–OH have been observed at 692 cm$^{-1}$ and 684 cm$^{-1}$ in MMT-2 and MMT-1 respectively. The Si–O band in MMT-2 and MMT-1 was observed to be shifted to a lower frequency at 1040 cm$^{-1}$ and 1039 cm$^{-1}$ respectively.

The adsorption of organic moiety causes Si–O bond polarization due to adsorption of positive charge on the silicate surface and thus the Si–O dipole moment and consequently the stretching band intensities were observed to increase in MMT-2 and CTAB–MMT, respectively.

The excessive nonstoichiometric amount of surfactant results in high accumulation of positive charge that provides the most intensive Si–O spectral bands.

Accordingly, the LCV molecules in MMT-3 are assumed to be attached to the Montmorillonite external face not only via the electrostatic forces related to the stoichiometric ion exchange but also due to interaction between aromatic nitrogen with the crystal edge or the other acidic site of MMT by electron transfer from nitrogen to MMT and/or hydrophobic π–π interactions of two neighboring aromatic rings.
The N-ring stretching mode is sensitive to changes in electron structure of the molecule. Indeed, when changing from the neutral molecule to the cation, its vibration frequency increases by 65 cm\(^{-1}\) from 1520 cm\(^{-1}\) in LCV to 1585 cm\(^{-1}\) in CV (crystal violet) as a result of the increased double-bond character. A small band at 1585 cm\(^{-1}\) has also been observed in MMT-3. The band at 1517 cm\(^{-1}\) is attributed to quinoid/semiquinoid ring vibrations, respectively [010, 011]. The H-O-H bending vibration band was found to shift towards shorter wavelength at 1630 cm\(^{-1}\) (from 1636 cm\(^{-1}\)) in MMT-3 and is accompanied by appreciable changes possibly due to the presence of LCV.

It can be observed from Figure 3.10B(d) that after the interaction of MMT to form a composite with chitosan, the O–H bending vibration of water in MMT at 1636 cm\(^{-1}\) was found to shift to a higher wavenumber at 1647 cm\(^{-1}\) in MMT-5 composites. The N–H bending band of chitosan at 1560 cm\(^{-1}\) is found to be shifted to a higher wavenumber at 1566 cm\(^{-1}\). These recommend some interaction between the entrapped water in MMT and N–H of chitosan. Also small shifts in Si–O in-plane stretching and C–O–C overlap at 1036 cm\(^{-1}\) have been observed that suggest the possible interactions. The O-H and N-H stretching bands at 3341 cm\(^{-1}\) (overlapping bands) that gives evidence of the presence of MMT in the intercalated composites. The FT-IR-ATR spectrum of CHITO-B indicates the presence of predominant bands at 2987 cm\(^{-1}\) and 2901 cm\(^{-1}\) (-CH stretching vibration in -CH and -CH\(_2\)), 1632 cm\(^{-1}\) (N-H bending vibration of protonated amino groups), the vibrational bands at 1148 cm\(^{-1}\) and 1033 cm\(^{-1}\) (C-O-C stretching vibration) in CHITO-B. The spectrum reveals that most of the functional groups originally present on chitosan are intact in CHITO-B even after formation of beads and are available for interaction with the metal ions [Table 3.2].

The FT-IR-ATR spectrum of MMT-4 shows broad O-H and N-H stretching vibration (overlapping) at 3324 cm\(^{-1}\). The O-H bending vibration at 1636 cm\(^{-1}\) has been found to shift to a higher wavenumber at 1645 cm\(^{-1}\). The vibrational band overlap at 1047 cm\(^{-1}\) (Si-O and C-O-C stretching) has been found to shift to a lower wavenumber at 996 cm\(^{-1}\).
Figure 3.10A. Fourier Transform - IR-ATR pattern of (a) MMT (b) MMT-1 (c) MMT-2
Figure 3.10B. Fourier Transform-IR-ATR pattern of (a) MMT-3 (b) CHITO-B (c) MMT-4 (d) MMT-5
### Table 3.2. Vibrational frequencies, cm\(^{-1}\), of modifiers and synthesized adsorbents

<table>
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<tr>
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<th>MMT</th>
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<th>CPC</th>
<th>LCV</th>
<th>CHI</th>
<th>TENTATIVE ASSIGNMENT</th>
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A: Pure modifier, B: modified MMT, C: MMT-4, D: CHITO-B

“υ” Indicates stretching, “δ” indicates bending
**Thermogravimetry (TGA – DTA)**

The thermal stability of MMT and synthesized adsorbents was analyzed using thermal studies (TGA-DTA).

The DTA curve of MMT shows weight loss in two parts (i) interstitial and surface adsorbed and free water (ii) interlayer water. The first step weight loss in the range from 20°C - 100°C (57°C) amounts to ~18% and is attributed to the volatilization of free/surface (physisorbed) water, the second step weight loss in the temperature range from 100°C - 250°C amounts to ~9%. The weight loss at 618°C owes to the loss of structural water resulting from the dehydroxylation of clay -OH units.

The thermogram of neat surfactant (CTAB, CPC) [Figure 3.11A] shows decomposition that begins at ~208°C. This weight loss corresponds to an exothermic peak at 350°C on the DTA curve. The TGA shows weight loss of ~71% that corresponds to the exothermic peaks in DTA leaving behind ~28% of residue indicating the formation of small amount of carbon.

The decomposition of modified Montmorillonite is found to take place in three steps: water desorption, modifier desorption and dehydroxylation of organoclay, as indicated by the thermograms [Figure 3.11A].

The CTAB modified MMT shows first step weight loss of ~4% in the temperature range from 20°C - 100°C that signifies less free water in MMT-1 due to occupancy of surfactant molecules and corresponds to an exotherm at 55°C in DTA. The MMT-1 shows weight loss within the temperature range from 160°C - 300°C corresponding to second step weight loss of ~4% and is attributed to the decomposition of surfactant physically absorbed on the external surface of MMT. The third weight loss of ~7.3% in the temperature range from 310°C - 450°C was observed corresponding to an endotherm at 350°C. MMT did not undergo thermally induced changes in this temperature range; hence the mass loss in this temperature range should be attributed to the decomposition of surfactant.
The CPC modified MMT shows first step weight loss of ~3% during the temperature range from 20°C - 100°C that signifies less free water in MMT-2 (than MMT) due to occupancy of CPC molecules [Figure 3.11A] and corresponds to an exotherm at ~55°C in DTA. The CPC-MMT shows second step weight loss of ~1% within the temperature range from 160°C to 250°C and is attributed to the decomposition of CPC physically adsorbed on the external surface of the MMT. The weight loss of ~18% in temperature range from 300°C - 450°C has been observed that corresponds to an endotherm at 356°C and can be attributed to the decomposition of surfactant.

A possible explanation is that the presence of surfactant lowers surface energy of the inorganic material and converts hydrophilic silicate surface to an organophilic [012].

The MMT-3 shows first weight loss of ~10% in the temperature range of 20°C to 100°C. This corresponds to a small endotherm at 56°C in DTA and is attributed to the decrease in physisorbed water as some LCV molecules may be attached to the surface of Montmorillonite. The weight loss of ~15% in the temperature range from 100°C - 250°C was observed corresponding to an exotherm at 179°C in DTA due to loss of adsorbed organic moiety [Figure 3.11A].

If the concentration of adsorbed moiety exceeds the CEC of clay, adsorbate molecules then adhere to the surface-adsorbed adsorbate ions by van der Waals forces [013]. Thus, the third step weight loss that corresponds to DTA peak at 356°C in MMT-1, 359°C in MMT-2 and 239°C in LCV-MMT is observed very close to the weight loss temperature of the pure adsorbate (CTAB =320°C, CPC =324°C and LCV =218°C respectively).

The first step weight loss in thermogram of CHITO-B was observed to be ~85·0 % in the temperature range from 25°C - 100°C that corresponds to an endotherm peak in DTA at 71°C due to the loss of water adsorbed both on the surface and in the pores of the beads [014]. The thermogram shows decomposition of chitosan beads in the second step weight loss of ~10·4% in the temperature range from 220°C - 420°C that corresponds to DTA peak at 280°C.
Figure 3.11A. TGA-DTA of various adsorbents
Figure 3.11B. TGA-DTA of various adsorbents
The CHI–MMT shows first step weight loss of ~9% that has been observed in the temperature range from 30°C - 100°C \[\text{Figure 3.11B}\]. This corresponds to an endotherm peak in DTA at 50°C resulting from the decrease in loss of water molecules than MMT possibly due to the occupancy of chitosan on MMT. The chitosan modified MMT was found to show second step weight loss of ~13% in the temperature range from 250°C - 300°C leaving behind ~80% residue. It can be observed from the thermogram that the degradation behavior of chitosan and its composites is different whereas MMT has been found to increase the thermal stability of the biopolymer/ clay composites. Thus, the addition of clay provides a thermal barrier, and hence delays the weight loss.

MMT-4 shows first step weight loss of ~6% in the temperature range of 25°C to 120°C, that corresponds to an endotherm at 58°C in DTA. The second step weight loss of ~48% from 120°C - 770°C was observed to be ~26% (130°C - 420°C) that corresponds to twin peaks at 288°C and 340°C in DTA and a weight loss of ~22% in the temperature range from 430°C - 800°C that corresponds to an endotherm peak at 602°C in DTA leaving behind ~44% residue.

- **Scanning Electron Microscopy**

The Scanning electron micrographs of the parent material, MMT and the corresponding synthesized adsorbents, MMT-1, MMT-2, MMT-3 and MMT-5 indicate a distinct change in surface morphology. Layered particles with equalized and smooth surface have been observed in MMT micrograph \[\text{Figure 3.12}\] whereas deformation of the smooth surface has been observed when MMT was interacted with the modifier. The surface of MMT-1, MMT-2 and LCV- MMT exhibits a grain like structure of agglomerated particles. The hydrophobized MMT particles can stack more easily than those of natural MMT because CTAB, CPC and LCV compensate for their negative charge and thus eliminate the repulsive electrostatic forces. The porous morphology of MMT-3 has been clearly observed as depicted in \text{Figure 3.12 (d)} suggesting significant
modification of MMT by LCV interaction and thereby a successful conversion of MMT into the modified MMT.

The micrograph of MMT-5 composite was found to be irregular and highly porous with network like morphology. It is believed that the formation of flocculated structure in MMT-5 biocomposites is due to the hydroxylated edge–edge interaction of the silicate layers. Since one chitosan unit possesses one amino and two hydroxyl functional groups that can form hydrogen bonds with the silicate hydroxylated edge groups which lead to strong interaction between matrix and silicate layers. This strong interaction is believed to be the main driving force for the assembly of MMT in the chitosan matrix to form flocculated structure. The results show that the use of chitosan promoted the clay disaggregation and thereby resulting in formation of highly porous sheet-like morphology. The CHITO-B and the MMT-4 were found to have a rough surface morphology and porous structure with pores of various sizes present on the surface as can be seen in the scanning electron micrographs.

Transmission Electron Microscopy

The Transmission electron micrograph of natural and synthesized adsorbents [Figure 3.13] shows well-defined spherical structure. The transformation of MMT to MMT-1, MMT-2, MMT-3 and MMT-5 shows the change in particle size as illustrated in the HRTEM micrograph [Figure 3.13]. The HRTEM of modified MMT shows a decrease in particle size with respect to MMT. The MMT-1 and MMT-2 shows diameter ranging from 0·008µm to 0·018µm and nm to 0·004µm to 0·014µm respectively and MMT-3 and MMT-5 ranges from 25nm to 44nm and 38·0 to 52·0nm respectively [Figure 3.13(d, e)].

The size of the spheres of CHITO-B and MMT-4 as observed on the basis of SEM analysis as TEM was required for size determination. The size of beads were found to be in the range of 1·0mm to 1·35mm and 1·1mm to 1·40mm for MMT-4 and CHITO-B respectively.
Figure 3.12. Scanning electron micrographs of (a) MMT (b) MMT-2 (c) MMT-1 (d) MMT-3 (e) CHITO-B (f) MMT-4 (g) MMT-5
Figure 3.13. Transmission electron micrographs of (a) MMT (b) MMT-1 (c) MMT-2 (d) MMT-3 (g) MMT-5


**Zetametry**

The zeta potential of the adsorbents as measured was observed to be +10·20mV for MMT. The modification of MMT with CTAB (surfactant) was observed to make the change in surface charge to increase by +16·7mV whereas CPC (surfactant) was observed to increase the surface charge to +21·0mV [Figure 3.14(a) and (b)].

The zeta potential of LCV modified MMT was observed to be +22·49mV. The modification of MMT with a biopolymer, chitosan was subjected to investigations of surface charge wherein the surface charge of MMT-5 was observed to be +36·30mV.

Thus, the modification of MMT with respective organic moieties (surfactant/s, leuco crystal violet and biopolymer) has been successfully performed and the surface charge of the adsorbent/s was observed to be beneficially made positive.

![Figure 3.14. Zeta potential of (a) MMT (b) MMT-1](image-url)
Figure 3.14. Zeta potential of (c) MMT-2 (d) MMT-3 (e) MMT-5
3.9 Conclusions

The modification of Montmorillonite, MMT has been performed successfully as depicted by the various characterization techniques of the synthesized adsorbents. The X-Ray Diffractograms show successful intercalation of MMT using CTAB, CPC, LCV and CHI as adsorbate/s that was depicted by the decrease in 2theta value and thereby an increase in d_{001} spacing of MMT after interaction.

The adsorbate was observed to move not only into the interlayer gallery but was also observed to show its characteristic bands on the surface as observed in Infrared spectrometry. This supports the presence of adsorbate/modifier (CTAB, CPC, LCV and chitosan) on the surface of MMT. The surface adsorption of the organic moieties is also supported by the scanning electron and transmission electron micrographs that show a distinct change in morphology of MMT on interaction with the respective moiety converting it into a porous and less aggregate form of MMT while the HRTEM marks a decrease in particle size of MMT after modification.

The thermal stability of the synthesized matrix had been analysed using TGA-DTA. The adsorbents were found to exhibit weight loss in the temperature range characteristic of the organic moiety that supports the presence of CTAB, CPC, LCV and CHI respectively on MMT.

The zeta potential values of the synthesized matrixes were found to exhibit a positive charge on the surface of adsorbents. Thus, MMT has been proved to be successfully modified by the respective modifiers as per the analytical techniques. The incorporation of characteristics of the modifier/s on MMT paved a path to the application of the adsorbents to be successfully used for the investigations of the extraction of toxic metal from aqueous solution.
References


