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Comprehensive Analysis of Surface Morphology and Elemental Composition Changes in Bioadsorbents Before and After Cocoon Adsorption

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Abstract

Adsorption processes have a wide range of applications in environmental science and materials research. Understanding the surface morphology and elemental composition of bioadsorbents is fundamental for assessing their effectiveness in these processes. In this study, we utilized advanced analytical techniques, including Scanning Electron Microscopy (SEM), Energy Dispersive X-ray Spectroscopy (EDS), and Fourier Transform Infrared Spectroscopy (FTIR), to investigate the structural and chemical changes that occur in bioadsorbents before and after cocoon adsorption. Raily cocoon, Daba BV, and Daba TV were selected as model bioadsorbents for this comprehensive analysis. This research paper investigates the surface morphology and chemical composition changes in tasar cocoons, specifically Raily cocoon, Daba BV, and Daba TV, before and after the adsorption process. The analysis was conducted using Scanning Electron Microscopy (SEM) to observe the morphological changes, and Energy Dispersive X-ray Spectroscopy (EDS) to determine the elemental composition. Furthermore, Fourier Transform Infrared Spectroscopy (FTIR) was employed to identify functional groups in the tasar cocoon components. The results reveal significant surface modifications and elemental interactions, shedding light on the adsorption capacity of tasar cocoons.

Keywords: *Daba, Raily, SEM, EDS, FTIR*

1. Introduction

Asia is the global leader in silk production, contributing 95% of the total output, with China and India being the primary producers. Japan, Brazil, and Korea also contribute. Sericulture, the practice of silkworm farming, plays a vital role in utilizing natural resources for socioeconomic development, providing livelihoods, employment, and income generation. In India, five main types of silk are produced: Mulberry Silk, Tropical Tasar Silk, and Temperate Tasar Silk. This industry has significant economic and social implications Nagaraju et, al and Malik MS et. al, (2008). Sericin, a natural polymer in silk, binds fibroin filaments, forming yarn. It's hydrophilic, with amino acids and polar groups. This allows it combine with other polymers, giving it unique properties like antioxidants, moisturization, antibacterial and UV protection, and possible antitumor effects. Silk cocoons comprise approximately 82% silk fibroin, 0.5% cocoon wax, 0.5% total ash, and around 2% minerals and natural colouring matter, primarily tannin (Komatshu et. al,1980). Notably, tropical Tasar silk cocoons possess exceptionally tough shells due to the presence of calcium oxalate and tannin. Unlike mulberry cocoons, which can be softened by plain water boiling using various devices, tropical Tasar cocoons require an alkaline medium with a pH exceeding 10 (Sonwalkar, 1993). The presence of calcium oxalate in the cocoon shell is stabilized by oxidative phenolic tannin, forming a dithrosine crosslinking, deposited during cocoon formation through caterpillar urine and faces (Gheysens et al., 2011). This inherent hardness and low non-broken filament length of tropical Tasar cocoons lead to challenges in the extraction process (Munshi et.al, 2015). In analyzing the data related to non-broken filament length (NBFL), it's observed that tropical Tasar cocoons exhibit positive skewness values, in contrast to the negative values found in mulberry varieties (Chattopadhyay et.al 2018). This skewness parameter accounts for the significantly higher reeling speed of mulberry cocoons, approximately 100 m/min using modern machinery, compared to the slower production rate of Tasar cocoons, approximately 30 m/min, primarily processed manually or with conventional machines (Sonwalkar, 1993). Tropical Tasar cocoons encompass 61 eco races, with 44 of them currently available. For yarn production via the reeling process, cocoons from eco races such as Daba, Raily, Modal, Sukinda, and Bhandara are commonly used. The Dabaecorace includes both domestic bi-voltine (BV) and tri voltine (TV) varieties, while others are bi-voltine (BV) sourced from natural resources (Jolly et.al, 1979). These variations in cocoon characteristics and processing requirements offer valuable insights into the challenges and unique qualities of Tasar silk production.

2. Literature Review

Zhang et. al (2013) explained that Tropical tasar cocoons are wild variety which has more compact structure than domesticated mulberry and semi domesticated muga (Antheraeaassamensis)cocoons due to higher inter fibre bonding length. FTIR- ATR spectra revealed the presence of calcium oxalate crystals in tropical tasar cocoons' outer layer. Interlaminar peel resistance and out of plane compressive modulus are higher for wild cocoons like Antheraeamylitta as well as Antheraeapernyi. Bombyxmoricocoon does not contain calcium oxalate crystal in outer layer. Gheysens et. al, (2011) discussed that, Demineralization enables elimination of minerals along with sericin from wild silk moth cocoons facilitates easy withdrawal of filament during reeling. Wild silk moth cocoons like Antheraeamylitta and Antheraeapernyi contain large quantum of calcium oxalate. Treatment using ethylene diamine tetra acetic acid (EDTA) leaves the gum substantially intact, preventing collapse and entanglement of the network offibroinbrins facilitates easy yarn extraction by wet reeling. Jena et. al (2018a & 2018b) done their research on Analysis of tasar silk sericin by FTIR spectra for structural determination revealed that presence of both α- helical and β- sheet in different eco races. Thermogravimetric estimation showed higher thermal stability and variable degradation profiles. The sericin was found to inhibit tyrosinase, elastase and glutathione-s-transferase activity and had apparent radical scavenging impact son 2.2 diphenyl 1 picrylhydrazil (DPPH), hydrogen peroxide and inhibition of lipid per oxidation Das, Kar, Chowdhury et. al (1994) told that Soaking in Biopril 50enzyme solution after steaming of tasar cocoons facilitates better softening and thereby more productivity with good yarn quality characteristics can be achieved. Shekar, Majhi et. al (1996), explained that Use of tamarind paste in de gumming solution with 1.5 to 2.0% concentration following 15 min boiling and60 min steaming helps for higher raw silk recovery. Das et. al, (1992) discussed about Wet reeling of tropical tasar cocoons provides higher yarn tenacity and cohesion along with better neatness and cleanness. Manna et. al, (1990), Similarly discussed that Biopril 50 and Anilozyme- Penzymes as well as raw papaya and raw pineapple extract improve the cooking efficiency of temperate tasar cocoons (oak tasar) with better productivity., Y.R., Singh & Devi et. al, (2012), analysed that, Higher cooking efficiency can be achieved by using pineapple extract with or without soda for temperate tasar cocoon. Lee et. al, (1999) determined that, for higher productivity of tasar silk filament, reeling speed need to enhanced. Among the different quality parameters, non- broken filament length (NBFL) is the most important characteristics for assessment of reeling performance. The reeling speed is directly proportional to the NBFL of silk cocoons. Das et.al, (2007) and Chattopadhyay et al, (2018) explained The mode values of NBFL exist I the lower range with respect to average values which is due to excessive breaks during withdrawal of filament and its statistical distributions are positively skewed different from normal distribution. Mitra et. al (2012), elucidated that the average NBFL ranges from 70 to 125 m for different varieties of tropical tasar cocoons whereas the average filament length varies from 300 to 400 m. Munshi et al, (2015), determined the higher reeling speed is very difficult to achieve and production is carried out at speed of about 25 m/min. Das at. al, 2005 and Divakara et. al, (2009) lower modulus along with higher extension of tasar silk filaments indicates poor orientation and crystalline order. Iizuka et. al, 1980, the segregation from Fillippi's gland of silkworm helps for better of liquid silk and thereby uniformity of filament. It is less active for non- mulberry silkworms as compared to mulberry. Chattopadhyay et al, 2018, hence there may be possibility for presence of more weak points throughout the progressive length of tasar silk filament. So, it cannot with stand high tension during reeling and thereby excessive breaks are occurring.

3. Materials And Methods

Scanning Electron Microscopy (SEM) Analysis:

Scanning Electron Microscopy (SEM) provides high-resolution images of surface structures. SEM was employed to capture the surface morphology of the selected bioadsorbents before and after cocoon adsorption. Obtain dried tasar cocoons, specifically Raily cocoon, Daba BV, and Daba TV, which will serve as the bioadsorbents in this study. Ensure that the cocoons are thoroughly cleaned and free from any external contaminants or impurities. Prepare samples of the dried tasar cocoons for SEM analysis. Mount the samples on SEM specimen stubs using a conductive adhesive to minimize charging during imaging. Conduct SEM analysis using a suitable SEM instrument to capture high-resolution images of the cocoon surfaces. Capture SEM images of the tasar cocoons both before and after the adsorption process.

Energy Dispersive X-ray Spectroscopy (EDS) Analysis:

Energy Dispersive X-ray Spectroscopy (EDS) was utilized to determine the elemental composition of the bioadsorbents' surfaces, shedding light on the chemical interactions associated with cocoon adsorption. Perform EDS analysis in conjunction with SEM to determine the elemental composition of the tasarcocoon surfaces. Carefully select specific regions of interest on the SEM images for EDS analysis to gather elemental data. Record the EDS spectra to identify the presence of elements such as carbon, oxygen, calcium, potassium, chlorine, aluminum, silicon, and any others that may be relevant to the study. Perform EDS analysis on tasar cocoons both before and after adsorption to compare elemental changes.

Fourier Transform Infrared Spectroscopy (FTIR) Analysis:

Fourier Transform Infrared Spectroscopy (FTIR) was applied to identify the presence of specific functional groups in the extracts of selected plant leaves, thereby confirming the chemical changes induced by cocoon adsorption. Obtain finely ground tasar cocoon samples for FTIR analysis. Prepare KBr pellets containing the tasar cocoon samples for FTIR spectra collection. Perform FTIR spectroscopy to record the infrared spectra of the tasar cocoon components. Analyze the FTIR spectra to identify specific functional groups and chemical bonds present in the tasar cocoon samples both before and after the adsorption process. Pay close attention to functional groups related to alcohols, phenols, N-H stretching, C=O stretching, aldehydes, nitro compounds, and other relevant features.

3. Results and Discussion

SEM & EDS Analysis: In this study, Scanning Electron Microscopy (SEM) to analyze the surface morphology of bioadsorbents, specifically Raily cocoon, Daba BV, and Daba TV, before and after the adsorption process. The SEM images revealed intriguing insights into the structural changes occurring on the surface of these tasar cocoons. Before adsorption, the SEM images (5.2SEMand EDX finding of cocoons before and after adsorption) depicted the dried cocoon surfaces as irregular in shape, characterized by a porous structure. This irregularity and porosity are key features that render tasar cocoons significant for adsorption purposes. Upon closer examination of SEM images after the adsorption process, remarkable modifications were observed. The surface area increased, and a peculiar thread-like dispersion emerged on the cocoon surface. Furthermore, the overall shape of the cocoons exhibited distortion, and the pores on the surface appeared to be occupied. These morphological transformations provide concrete evidence of adsorption occurring on the surface layer of the tasar cocoons. The Energy Dispersive X-ray Spectroscopy (EDS) analysis further unveiled the elemental composition of the cocoon surface. Notable elements such as carbon, oxygen, calcium, potassium, chlorine, aluminum, and silicon were detected on the surface, indicating an intricate interplay of these elements. This elemental analysis confirmed the presence of chemical interactions taking place on the surface of the tasar cocoons. In summary, SEM analysis highlighted the significant changes in surface morphology before and after adsorption, including increased surface area, thread-like dispersions, shape distortion, and pore occupation.

SEM and EDX finding of cocoons before and after adsorption-

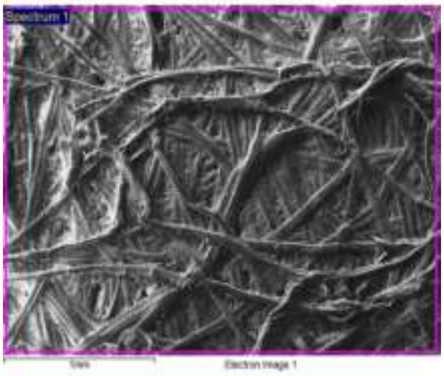

Fig. 5.2 a. RAILY COCOON Before Adsorption

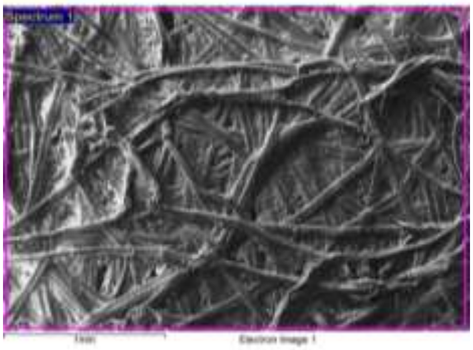

Fig. 5.2 b. RAILY COCOON Before Adsorption

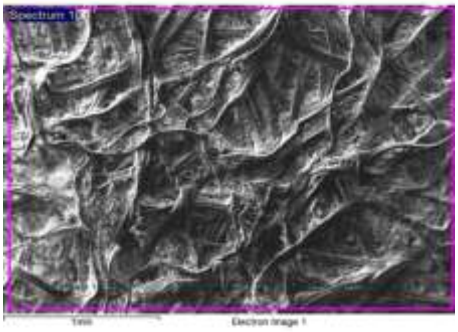

Fig. 5.2 c. DABA BV Before Adsorption

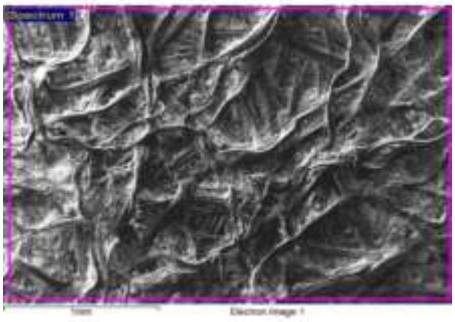

Fig. 5.2 d. DABA BV After Adsorption

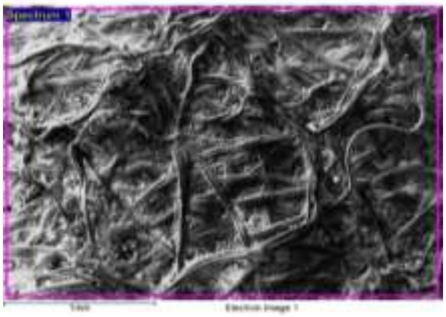

Fig. 5.2 e. DABA TV Before Adsorption

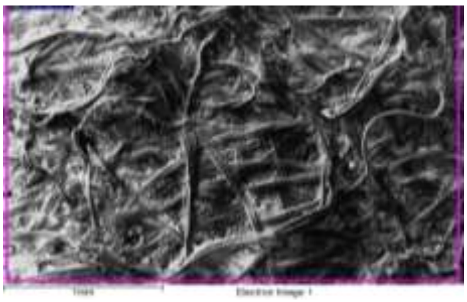

Fig. 5.2 f. DABA TV After Adsorption

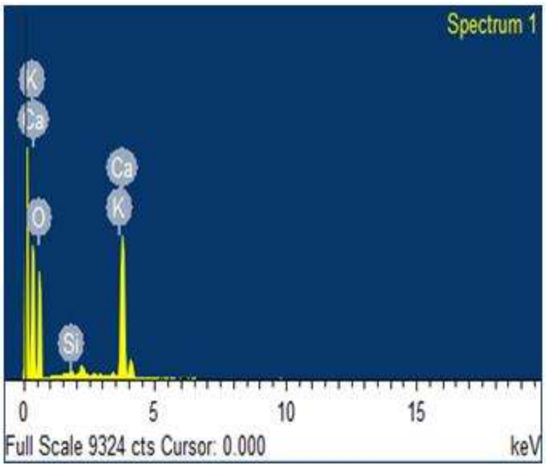

Fig. 5.2 (1) EDS of before DABATV

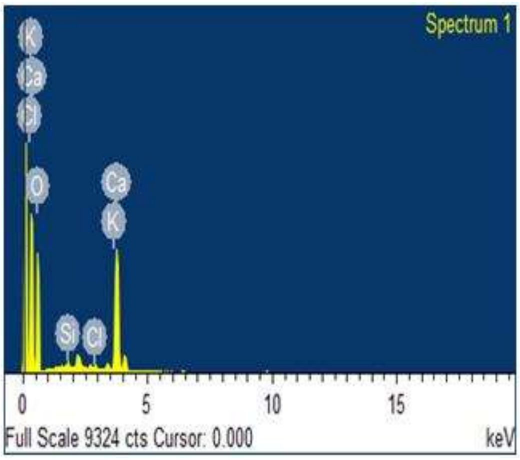

Fig. 5.2 (2)EDS of after DABATV

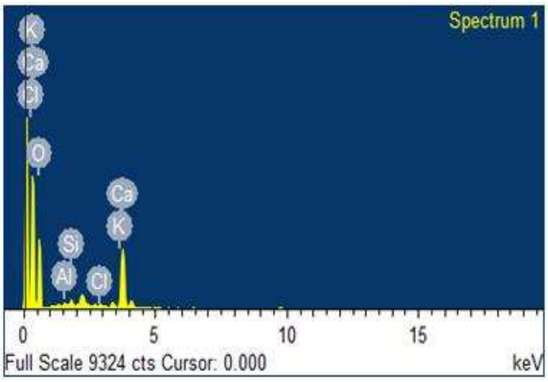

Fig. 5.2 (3)EDS of before DABABV

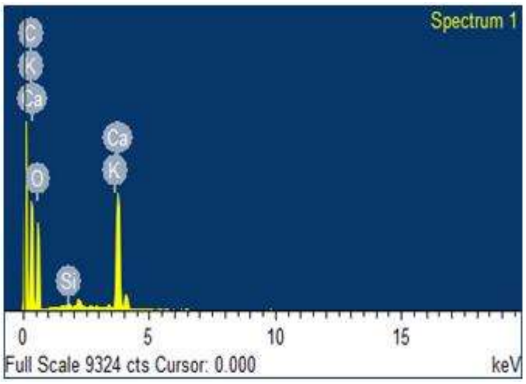

Fig. 5.2 (4) EDS of after DABABV

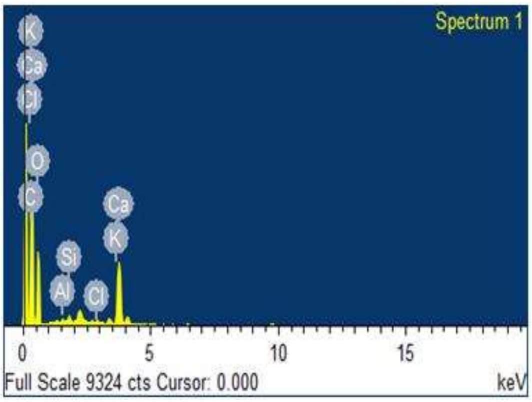

Fig. 5.2 (5) EDS of before RAILYCOCOON

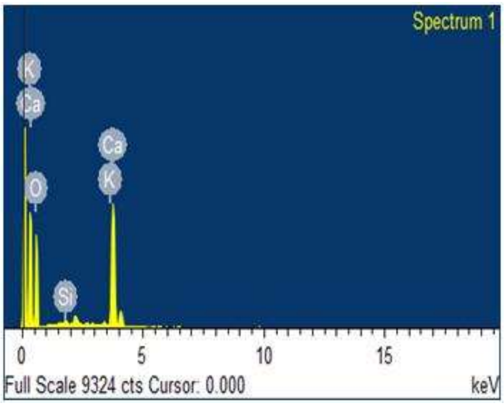

Fig. 5.2 (6)EDS of after RAILYCOCOON

These observations provided conclusive evidence of adsorption taking place on the tasar cocoon surface. Moreover, EDS analysis revealed the elemental diversity on the surface, substantiating the complex chemical interactions involved in this process. Together, these findings emphasize the potential of tasar cocoons as effective bio adsorbents for a wide range of applications, particularly in the domain of adsorption processes. EDS analysis revealed the presence of several elements on the cocoon surfaces. These elements included carbon, oxygen, calcium, potassium, chlorine, aluminum, and silicon. These findings indicated that chemical interactions between the bio adsorbents and the adsorbate, tasar cocoons in this case.

FTIR Spectroscopy:

The FTIR spectroscopy approach was used to identify the functional groups contained in active components and was based on the peak values observed in the infrared region radiation. The FTIR spectrum demonstrates the presence of characteristic stasarcocoons components in plant leaf extracts. The peak ratio in the spectrum was used to separate the functional groupings. In the present study 1F(before-after), FTIR spectrum obtained for the Raily cocoon shows the existence of extensive range of functional groups determined FTIR spectra analyses confirm the existence of alcohols and phenols having a value of peak at 3564.33cm^{-1} which corresponds to O-H stretching, the peak at 3349.98cm^{-1}, 3058.04cm^{-1} which have strong N-H stretching, 1739.41 cm^{-1} C=O stretching aldehyde,1690.86 C=N Oxime, 1551.04 N-O nitro compound, 1369.87 O-H bending alcohol, 810.67 C=C bending, 761.80 C-H bending , 664.26 C=C strong bending , 612.09 –C=C–H bend alkynes, 538.64 C–Br stretch alkyl halides.

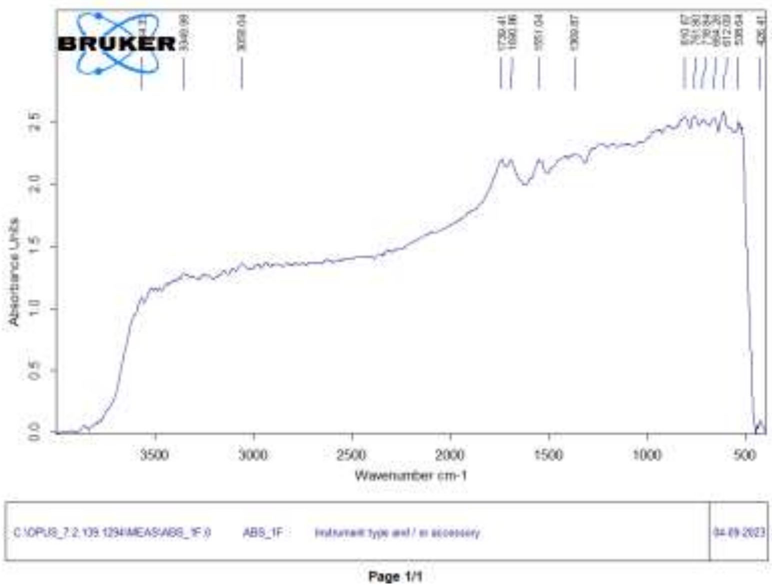

Fig-5.3 a. FTIR RAILY COCOON BEFORE Adsorption

AFTER Adsorption 3565.96 corresponds to O-H stretching, 3465.21cm-1-3461.42 cm-1 O-H stretch, H-bonded alcohols, phenols, 3350.13N-H stretch 1°, 2° amines, 3095.28 C-H stretch aromatics, 2991.43 C-H stretching alkanes, 2328.01cm-1 C-H stretching aldehydes, 2108.

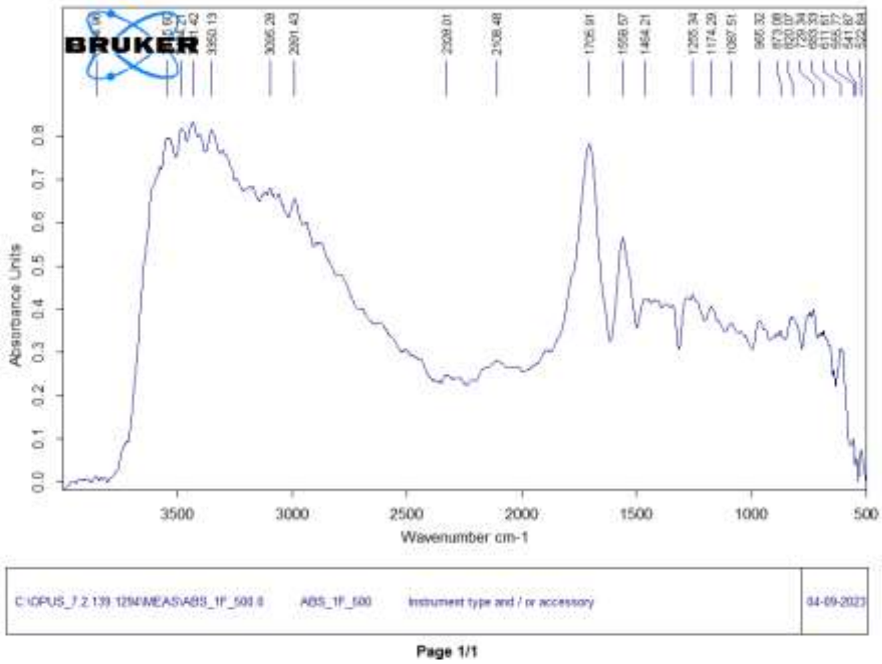

Fig-5.3 b. FTIR RAILY COCOON AFTER Adsorption

In the present study, FTIR spectrum obtained for the Daba BV cocoon shows the existence of extensive range of functional groups determined were illustrated in fig FTIR spectra analyses confirm the existence 3549.97, 3325.32-,3209.62cm^{-1}O–H stretch, H–bonded alcohols, phenols, ,3151.81,3069.84cm-1 C–H stretch aromatics,2924.81-2855.90cm-1C–H stretch alkanes.

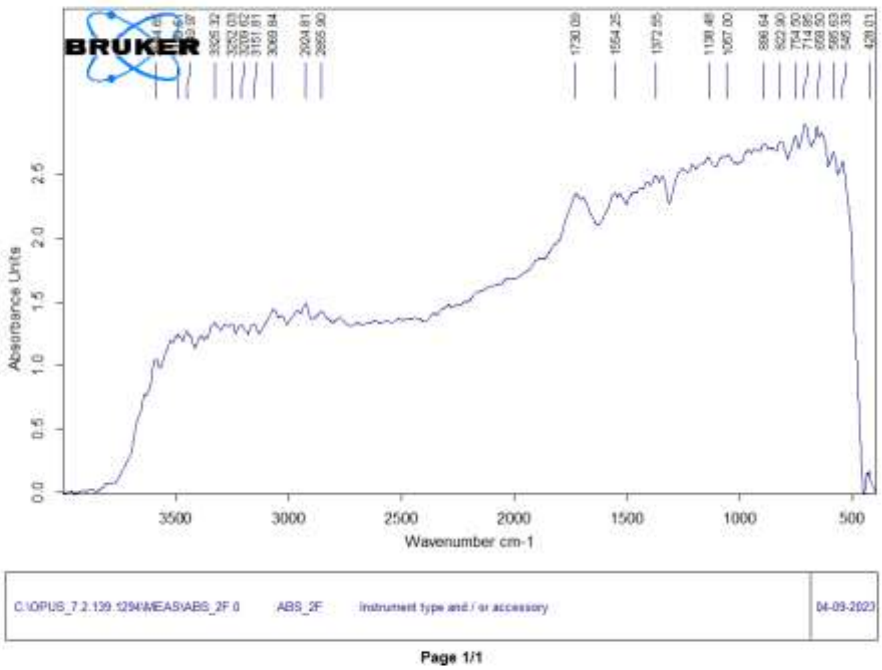

Fig-5.3 c. FTIRDABA BV COCOON BEFORE Adsorption

After adsorption, 3464.21cm-1, 3408cm-1 O-H stretching free hydroxyl alcohols, phenols, 3365 cm-1, 3331cm-1 3266cm-1 N-H stretching primary secondary amines, 3223cm-1 C-H stretch alkynes, 3120 cm-1 C-H stretch aromatics, 3084cm-1 =C-H stretch alkenes, 2947.19cm-1-2857.92 cm-1 C Stretch aromatic amine, 951.75 cm-1 =C-H bend alkenes.848.11N-H wag 1°,2° amines, 720.68 cm-1 621.97 cm-1 C-Cl stretch alkyl halides 554.43 cm-1 C-Br stretch alkyl haides.

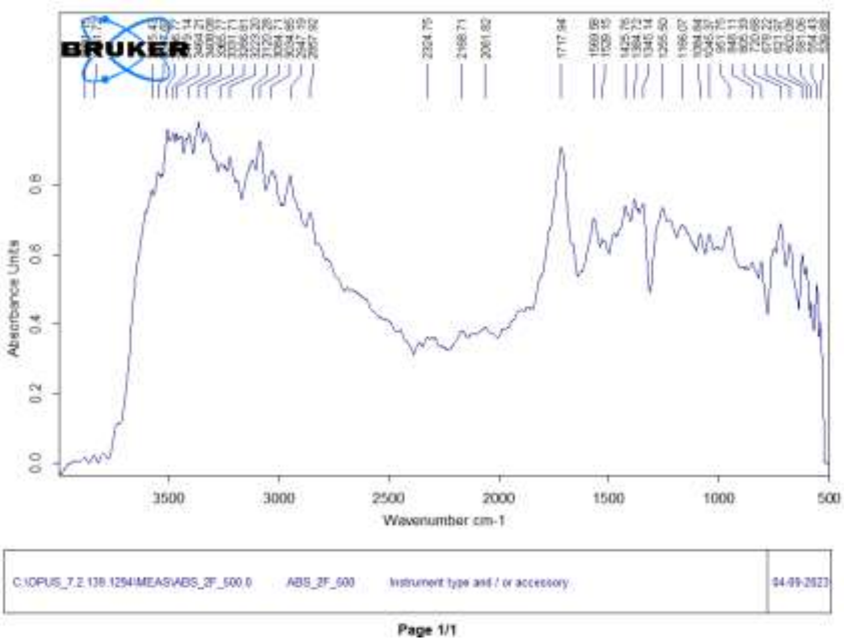

Fig-5.3 d. FTIR DABA BV COCOON after Adsorption

In the present study, 3325.78 cm-1 N-H stretch primary secondary amines and 3240.79 cm-1 O-H stretch 3058.74 cm-1 =C-H stretch alkenes, 2923.91 cm-1 C-H stretch alkanes, 1716.93 cm-1 C=O stretch alpha beta -unsaturated aldehydes., 1461.85 C-C stretch aromatics, 1345.34 cm-1 N-O symmetric stretch aromatics amines, 815.79 -592.80 cm-1 C-Cl stretch aliphatic amines, 532.

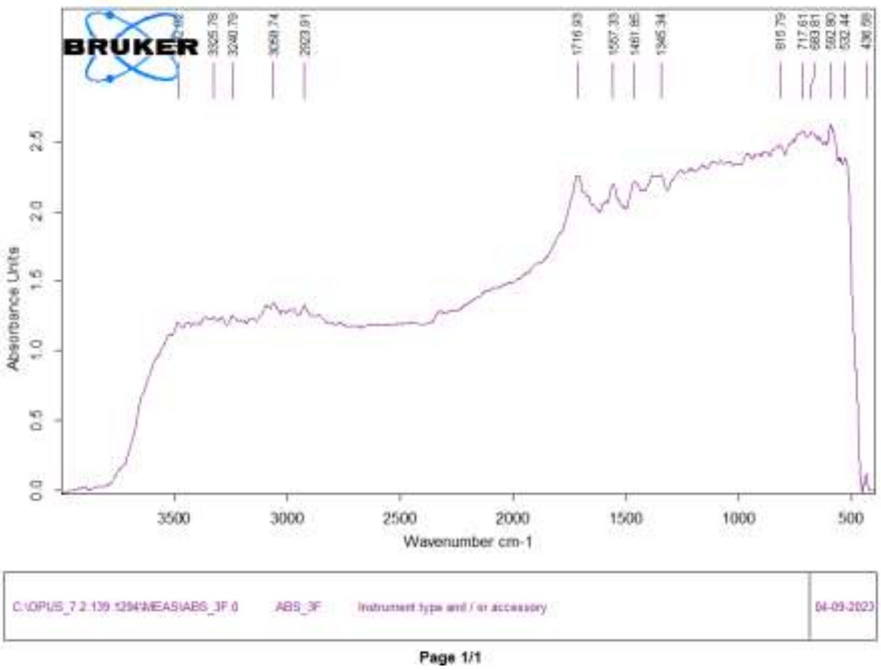

Fig-5.3 e. FTIR DABA TV COCOON before Adsorption

This study examined primary secondary amines with N-H stretch measurements of 3327.32 cm-1, O-H stretch measurements of 3268.28 cm-1, and H-bonded alcohols, phenols, 3170.51 cm-1, 3073.39-2853.09 cm-1, O-H stretch carboxylic acids, 2132.54 cm-1, C=C-stretch alkynes, 1704.87 cm-1, C=O stretch carbonyls, 1453.57 cm-1, C-H bend alkanes, 1244.88 cm-1, - 1006.73 cm-1, C-N stretch aliphatic amines, 963.44 cm-1, - 611.42 cm-1, O-H bend alkenes, 560.56 cm-1, -538.18 cm-1, C-Br stretch alkyl halides.

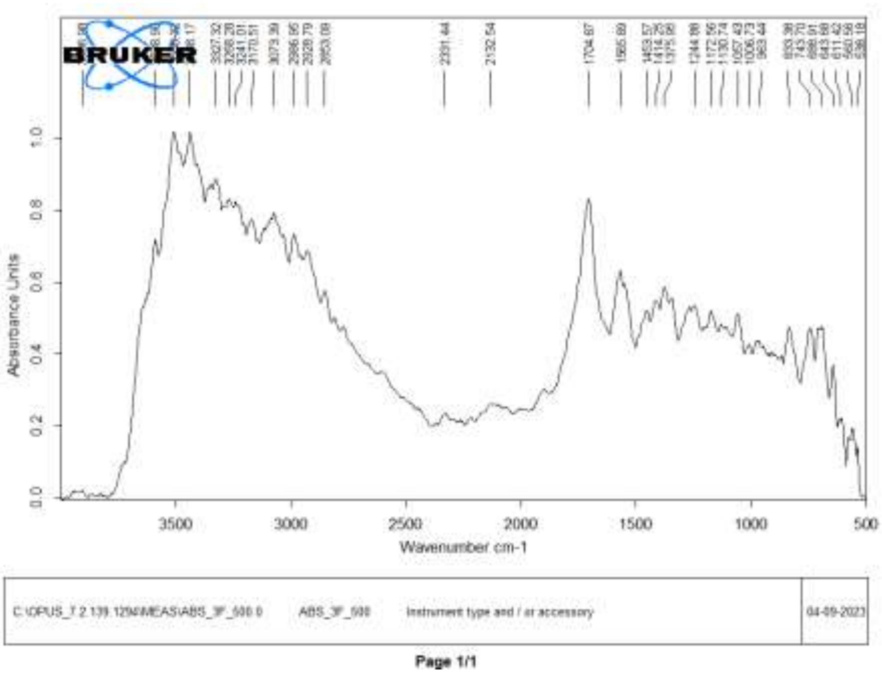

Fig-5.3 f. FTIR DABA TV COCOON AFTER Adsorption

4. Conclusion

The combined use of Scanning Electron Microscopy (SEM), Energy Dispersive X-ray Spectroscopy (EDS), and Fourier Transform Infrared Spectroscopy (FTIR) has shed light on the surface morphology and chemical transformations occurring in tasar cocoons, specifically Raily cocoon and Daba BV, before and after the adsorption process. The comprehensive insights obtained from these three analytical techniques enable a robust comparative conclusion. SEM images depicted the surface of dried tasar cocoons as irregular in shape and porous before adsorption. This irregularity and porosity serve as essential attributes for adsorption processes. After adsorption, SEM revealed significant surface modifications. Notable changes included an increase in surface area, the emergence of thread-like dispersions, distortion in shape, and occupation of pores. These morphological changes strongly substantiate the occurrence of adsorption on the surface layer of the tasarcocoons. EDS analysis unveiled the elemental composition on the surface of the tasar cocoons, identifying elements such as carbon, oxygen, calcium, potassium, chlorine, aluminium, and silicon. The presence of these elements indicates complex chemical interactions taking place on the cocoon surface. FTIR spectroscopy allowed for the identification and characterization of functional groups present in tasarcocoons. The FTIR spectra revealed a wide range of functional groups, including alcohols, phenols, N-H stretching, C=O stretching, and various others. After adsorption, significant shifts in peak positions were observed, indicating changes in functional groups and chemical bonds on the cocoon surface. The comparative analysis of SEM, EDS, and FTIR results provides a holistic understanding of the surface modifications and chemical interactions occurring in tasar cocoons during the adsorption process. Notably, SEM visualizations confirmed the substantial changes in surface morphology, with increased surface area and occupation of pores, reinforcing the adsorption capacity of tasar cocoons. The EDS analysis further confirmed the presence of various elements on the surface, substantiating the chemical complexity of the adsorption process. The FTIR analysis, on the other hand, delved into the chemical composition and functional groups present in the tasar cocoons. Shifts in peak positions and the emergence of new functional groups after adsorption clearly indicated alterations at the molecular level. These changes point to the dynamic nature of bioadsorption on the tasar cocoon surface. In conclusion, the integration of SEM, EDS, and FTIR analyses has provided comprehensive evidence of the effectiveness of tasar cocoons as bioadsorbents. These findings not only advance our understanding of the adsorption capacity of tasar cocoons but also hold promise for their applications in diverse environmental and industrial contexts. The observed surface modifications and chemical transformations underscore the potential of tasar cocoons as sustainable solutions for addressing various challenges through adsorption processes.

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