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GREEN COMPOSITE FILM FOR FOOD PACKAGING APPLICATIONS

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Abstract

In this study, isolation and characterization of nano cellulose from peanut shell was carried out. A new biodegradable antimicrobial film was developed using chitosan, zero valent iron (zvi) nanoparticle and nanocellulose. Nano cellulose got from peanut shells was utilized as filler and iron oxide as hostile to microbial reinforcer for chitosan. Another arrangement of green composite film was additionally arranged by surface alteration of nano cellulose and tried for antimicrobial property. The two films were described utilizing Fourier transform infrared (FTIR) spectroscope, X-ray diffraction (XRD) technique, Scanning electron microscope (SEM), thermo gravimetric analysis (TGA) and contact angle estimations. Antibacterial activity of the prepared green composite films was directed against Staphylococcus aureus ATCC 25923 and Escherichia coli ATCC 25922 by agar dispersion strategy. The outcomes demonstrated that Peanut shell waste could turn into a suitable wellspring of monetarily significant nano cellulose. It was likewise discovered that cellulose got from Peanut shells improved mechanical properties like tensile and impact tests. The high degradation temperature of nano composite showed prevalent thermal stability. All the characterisation underlined that carboxy methylated nanocellulose fortified compososite film displayed more sensitivity and can be viably utilized as packaging material to improve the shell life of food materials.

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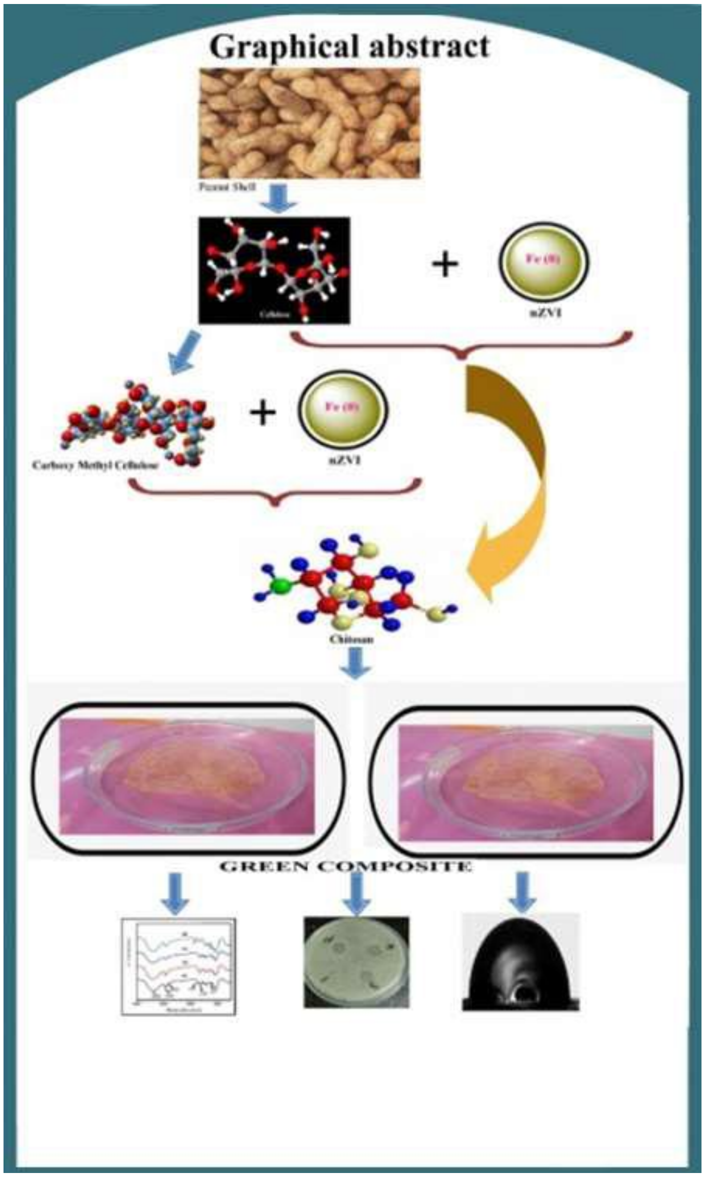

1. Introduction

Biopolymers are essentially utilized and depended in food packaging industry to diminish the profundity, rate and spread of ecological issues. The materials got from the plants, creature products and microbial products are called biopolymers. Cellulose is the main accessible polymer joined by strength and stiffness on the planet (1, 2). Cellulose is found with hemicelluloses, waxed resins, lignin etc. (3) Cellulose is derived from the plant assets, for example, wood, cotton, sisal, coconut fiber and from microscopic organisms, tunicates and so forth, and the resources other than the plants. Its structure is as fibrils in lignin network. Cellulose is differently utilized in light of the fact that there are three hydroxyl groups in it which can create hydrogen bonds (4, 5).

Be that as it may, films delivered by such cellulose have certain drawbacks of mechanical property (6). The disadvantages of such mechanical execution are rewarded by including biodegradable polymers and fillers. Packaging industry gives incredible criticalness to nano sized cellulose since, when plainly visible materials are utilized as fortify it shows poor adhesion character with network (7). The cellulose with nano size gives high perspective proportion, more than the ones lacking nano size. So this cellulose gives brilliant fortifying impact in polymer grid with nano particles having high quality and film straightforwardness (8). Polysaccharides, lipids and proteins are the customary biodegradable polymers with got from plants and creatures. The film industry chooses starch, chitosan and so forth., as biodegradable polymers since they are less expensive as well as effectively accessible. Among these chitosan is noteworthy in light of the fact that it can create incredible non-toxic films. Other than the films with chitosan have exceptional antimicrobial property against microbes, yeast and mold (9).

When the nano filler is additionally added to the polymer network, the physic-chemical property will be a matchless one. This improves primarily the quality and antimicrobial limit (10-17). Water retention is another quality or idiosyncrasy of packaging film. It shows the expanding limit of packaging film when it interacts with food and fluid. At the point when such properties are estimated, one can see how the packaging film acts in various conditions (18). Cellulose can be utilized in various structures. It very well may be utilized as unique fiber, methyl cellulose or carboxy methyl cellulose. Right now, film dependent on chitosan, cellulose separated from nut shells and iron nano particles were utilized. In addition, a similar cellulose which is altered to carboxy methyl structure based film was additionally studied for packaging application.

2. Materials and Methods

2.1. Materials

Peanut shells acquired from a nearby plant close Palakkad were utilized as the crude material. Synthetic compounds utilized were NaOH (Merck), NaOCl, HNO3 and Ethanol. All synthetic concoctions utilized right now of high virtue and of investigative reagent (AR) grade and were utilized as gotten. Twofold Distilled Water (DDW) was utilized all through the examination. Chitosan ≥75% (deacetylated) bought from Sigma Aldrich. Agar arrangements were acquired of Mueller-Hinton. Eschericha coli ATCC 25922 and Mangifera indica extricate were given by Adson chemicals.

2.2. Methods

2.2.1. Isolation of micro crystalline cellulose from peanut shells

Figure 1 (a). Pure Peanut shell powder (b) alkali treated (c) HNO3/ ethanol refluxed (d) Micro crystalline cellulose.

The groundnut shell was granulated into crystalline powder and dried for a time of one week. It was then sieved to acquire finely crushed powder. A cartridge containing 50g of dried groundnut shell was set in a Soxhlet outfitted with 1000 mL R.B flask. The example was first exposed to extraction at 85°C with 600 mL ethanol/toluene (2/1 (v/v)) blend during 24h [19]. The pulping procedure was performed under barometrical tension. The acquired blend was then quickly separated. The residue was progressively washed with 350mL of NaOCl solution (40 wt%) for 18h at 30°C, 250mL of ethanol for 2h at 30°C and 250mL of diethyl ether for 2h at ordinary temperature [20]. After this treatment, the cellulose pulp was washed with distilled water until pH of 7 was come to. Finally, the residue acquired was dried for 24h at 60°C.

In the preparation, 100mL HCl solution was taken in a 250mL cup and kept it in a warming mental. The temperature of warming mental was kept steady at 85°C and stirring of HCl solution began. In the wake of getting the 85°C temperature of HCl arrangement, 100 g of the groundnut shell pulp were included the solution gradually with persistent stirring. After complete addition of the powder, the mixture was held under continuous mixing for one and half hours. Subsequent to affirming the total de polymerization of the powder, the heating and stirring was stopped and the blend was saved for cooling. Subsequent to getting the blend at room temperature, it was neutralized with water to get the neutral pH. At that point this blend was filtered with the Watmann 42 filter paper isolating microcrystalline cellulose (MCC). At that point this microcrystalline cellulose was again washed with acetone to expel hints of water present. Again filtration was done to isolate acetone and microcrystalline cellulose. After filtration, the microcrystalline cellulose was saved for regular drying. After complete drying, the microcrystalline cellulose was gauged (21, 22).

2.2.2. Preparation of Nano Cellulose from Micro crystalline cellulose

About 5g of MCC (separated from nut shell) was blended in with sulphuric acid (64wt%, 45ml) kept in an ice shower, the blend was mixed for 120 min at 450 0C utilizing a magnetic stirrer. Around 500 ml of water was added to the reaction mixture to stop hydrolysis and afterward washed till the arrangement got neutral. The resultant suspension was saved for freeze-drying.

2.2.3. Conversion of isolated cellulose into CMC

Separated cellulose from peanut shell waste was changed over to CMC in two stages: alkalization and etherification of cellulose under heterogeneous conditions. In the initial step about 5.0g of isolated cellulose powder was gauged and added to 500mL Schott bottle and followed by 300mL of water: isopropyl alcohol solvent (1.4) in fitting proportion. At that point, 40mL of different Concentrations (5, 10, 15, 20, 25 and 30%)w/v sodium hydroxide included drop-wise and mixed for 2 h. The carboxy methylation reaction was begun by including varying measures of sodium mono chloro acetic acid (MCA) (1, 2, 3, 4 and 5g) to the reaction mixture on magnetic stirrer hot plate. The reaction mixture was warmed up at different temperatures (40-

60⁰C) with steady mixing for 3h. The blend was then separated and the residue was suspended in 100mL of methanol for 40 min. The suspended slurry was then neutralized with dilglacial acetic acid. The residue was filtered again and washed with methanol. The buildup from the filtration was dried in hot oven at 60⁰C overnight and the powder acquired was CMC (23).

2.2.4. Film Preparation with Iron nanoparticles

Polymer solutions and the nano particle solutions were arranged independently (18). The two solutions were blended at room temperature under constant stirring. Then again, solutions from silver nano particles (0, 0, 05, and 0, 5% w/v) were set up in distilled water and ultra sonicated for 30 min. These solutions were added to the polymer solutions recently arranged and were exposed to a solid mixing for 2 hours at 70°C. The solutions were casted in glass Petri dishes and dried in a heater at 45°C for 24 hours. Finally, the dried films with a thickness of ~3 mm were gotten and put away at consistent temperature in a desiccator for 48 h to be utilized later.

2.3. Characterisation

Scanning Electron Microscope (SEM) Analysis

The morphology of the readied composite when treatment were contemplated utilizing SEM (TESCAN vega).

Fourier Transform Infrared Spectrometer (FTIR)

The composite were clarified utilizing Fourier Transform Infrared Spectrometer (FT/IR) (Jasco 460 or more model). The FT-IR spectra for composite were gotten over the frequency running from 500 to 4000cm−1 utilizing potassium bromide disc technique.

X-ray diffraction technique (XRD)

The structure and period of the integrated composite were portrayed by XRD (Bruker parallel v3 X-ray diffractometer) wiyh Cu kα (λ = 1.5406 A0) radiation as the X-ray source worked at 40 Kv and 30 Ma. The patterns were gathered in the scope of2θ =10-800.

Thermo gravimetric Analysis

A thermo gravimetric analyzer (SEIKO model TG/DTA 6200) was used to quantify the thermal weight reduction and derivate thermo gravimetric of the film in the temperature extend somewhere in the range of 30^0C and 600^0C, with a warming pace of 100C/min under nitrogen stream. The weight loss at various stages were checked.

Anti bacterial test

Antimicrobial activity test of film was completed utilizing agar diffusion strategy. The films were cut into a plate structure. Film cuts were put on Mueller Hinton agar plates which had been recently seeded with 0.1 ml of inoculums containing pointer micro organisms. The plates were then hatched at 370C for 24h. The breadth of inhibitory zone encompassing film circles of films with agar surface was then estimated.

3. Results and Discussion

3.1. Fourier transform infrared spectroscopy (FT-IR)

FTIR spectra of untreated PSP, soluble base treated, ethanol refluxed and MCC are introduced in Figure 2. Contrast in intensities of FTIR groups demonstrated that changes have happened during chemical treatment. All the spectra has broad band in the locale of 3400cm-1, 2900 cm-1 and 1627 cm-1. The one at 3400 cm-1 compares to the free O-H extending vibration of -OH in cellulose. Those at 2900 cm-1 and 1627 cm-1 were appointed to C-H stretching vibration (24) and O-H bending of consumed water (25). Pure PSP demonstrated strong band at 1735 cm-1 and 1247 cm-1(26), comparing to C=O stretching vibration of acetyl groups from lignin and hemicelluloses; and C-O out of plane extending vibration of phenyl group in lignin. These two groups progressively vanished during chemical treatment. The nonappearance of these two groups in MCC demonstrates the total expulsion of lignin and hemicelluloses (27).

The FT-IR Spectra of CMC (Fig. 3) are like the spectra revealed already (28). The FT-IR spectrum of the example demonstrated that the carboxy methyl and hydroxyl groups are found at frequency of 1620, 1423 and 1328 cm-1 respectively. Clearly the expansive retention band at 3423 cm-1 is because of stretching hydroxyl group. The band at 2920 cm-1 is because of carbon-hydrogen (C-H) stretching vibration. The strong absorption at 1620 cm-1, affirmed the nearness of COO− group. The groups around 1423 and1328 cm-1 are due to -CH 2 scissoring and hydroxyl groups bending vibration, individually. The band at 1054 cm-1is expected to>CH-O-CH 2 stretching. The top at 1600-1640 cm-1 and 1400-1450 cm-1 is because of the carboxyl and its salts, which is affirmed substitution of carboxy methyl groups in cellulose structure. This peak doesn't exist in the FTIR spectra of cellulose from nut shell acquired (29).

The C-H extending band and C-N stretching band are seen at 2870 cm-1, and 1253 cm-1 respectively. These groups are available in secondary amines, and have a place with the chitosan polymer chain. The noticeable peak that showed up at 1634 cm-1 was appointed to both the carbonyl extending of the secondary (amide II) groups of chitosan (30, 31). Fourier Transform Infrared Spectroscopy (FTIR) demonstrates chemical changes in the polymer structure. Cellulose and carboxy methyl have comparative functional groups with same absorption.

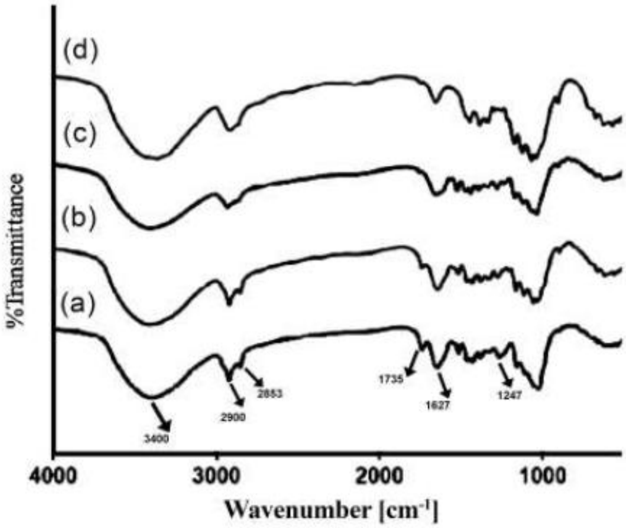

Figure 2. FTIR spectra of (a) untreated PSP (b) alkali treated (c) acid refluxed (d) MCC.

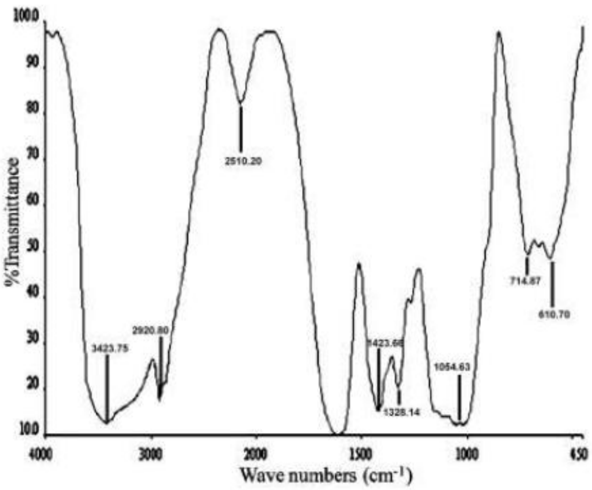

Figure 3. FT-IR spectra of CMC.

FTIR spectrum presents two new peaks located at 546 and 634 cm-1 related to stretching vibration of Fe–O. Meanwhile, the band at 3039-3710 cm-1 assigned to the stretching vibrations of hydroxyl groups, shifts from 3444 cm-1 to lower wave number 3423 cm-1, suggesting the strong interaction between the hydroxyl groups of cellulose and the encapsulated Fe $_2O_3$. From the above, the Fe $_2O_3$ nano particles were successfully fabricated and encapsulated in the architectures of cellulose (32)

3.2. X-ray diffraction (XRD)

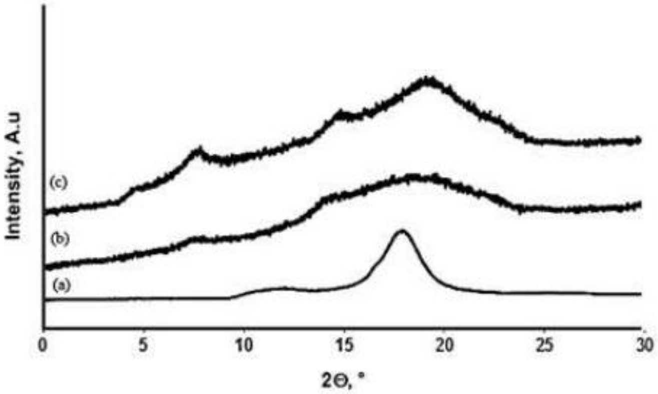

Figure 4. X-ray diffractograms of films based on (a) NCC, (b) chitosan and (c) chitosan+ NCC.

Structural examination of chitosan films and NCC containing chitosan films was explored by XRD. Fig.4 speaks to the diffractograms of the pure chitosan film and the NCC fortified chitosan films. The diffractogram of NCC films displayed crystalline at 2θ = 16.80 and 20-22.40. The watched peaks might be because of the 1 10 and 2 2 0 planes of cellulose I, separately. (33). Chitosan films indicated a trademark sharp peak at around 2θ = 130 and an broad one at 2θ = 20-230. The sharp one at 2θ = 130 demonstrated a hydrated crystalline structure, while the broad one showed an indistinct structure of chitosan (34, 35). Because of the fuse of NCC into chitosan, an expansion in top force at 2θ = 130 was watched. Likewise, the broad one of indistinct chitosan at 2θ = 20–230 super-positioned with the sharp peak of NCC at 2θ = 220. A sharp one at 2θ = 250 was watched for the chitosan with NCC. This diffractograms recommended that NCC strengthened chitosan films showed a blend of indistinct and crystalline peaks (36). The expansion of intensity of the chitosan films may result from the trans crystallization impact. Transcrystallization can be characterized as direction of crystals of a semi crystalline framework oppositely to the cellulose nano crystals (37) announced crystallization of polymer network specially nucleated by cellulose nanocrystals, prompting a trans crystalline layer around the nano crystals. The improvement of boundary properties of NCC-strengthened chitosan films may be ascribed to the nearness of crystalline locales in the films. Higher the level of crystallinity, bring down the porousness of the films (38). At last, XRD bolstered both the mechanical and barrier property which improve chitosan films. It was noticed that other than cellulose and chitosan, the composite films showed some unmistakable peaks at 2θ estimations of 30.3, 35.7, 43.5, 53.7, 57.2 and 63.0, which were related to the trademark peaks of Fe 2O 3(39)

3.3. Thermo gravimetric analysis (TGA)

Figure 5 portrayed the TGA and DTG bends, separately. All the TGA bends started with slight mass losses from room temperature to 1050C, relating to the dissipation of somewhat bound water from all the samples. As the temperature expanded further, the degradation happened in two stages. The main disintegration happened between 2140C and 300 0C, comparing to the hemi cellulose degradation and the start of lignin debasement (40, 41). After this change, the biggest loss of mass in the material happened between

3000C and 3800C, topping at 3420C, which compared to that of cellulose (42). TGA for the films were spoken to were truly steady during the heating of 50-2750C, losing just 4.2% of its underlying weight. The weight reduction for the chitosan, chitosan with NC was 26.7, 27.6 and 27.8%, individually. For both the films a significant weight reduction was found at around 2800C which is related to quick volatilization of polymer portions because of scission of the polymer spine. Chitosan showed a comparable stability at the temperature extend 280-4600C and the impact of NC on the thermal stability was seen as unimportant.

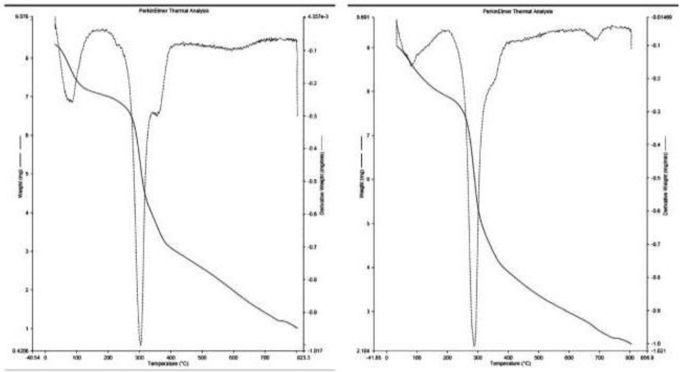

Figure 5. TG patterns and corresponding DTGA analysis.

3.4. SEM analysis

Figure 6a shows SEM analysis of NCC. chitosan embedding cellulose fibers were shown in figure 6b. Aggregates on top of the chitosan in the case of chitosan-cellulose-nzvicomposite, and smaller and dispersed aggregates in the case of chitosan-CMC-nzvi composite (Figure 6c).

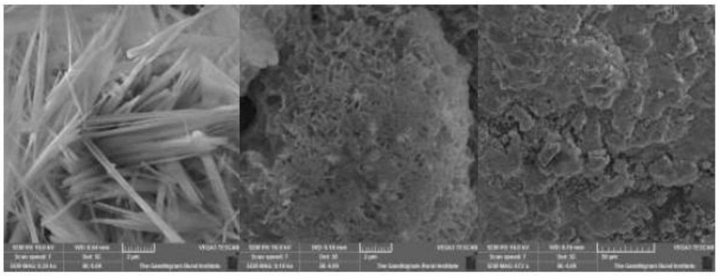

Figure 6(a). SEM micrograph images of NCC b) cellulose composite c) CMC composite.

3.5. Contact angle measurements

The contact angle is a wetability of a surface by a liquid. It extensively characterizes the hydrophilic/hydrophobic character of the surface. The contact angle consequences of the composites are appeared in Figure 7. The contact angle degrees are 850, and 1020 for cellulose based composite and CMC composites separately. The contact angle is lower for cellulose composite than altered films. NCC/chitosan/Fe2O3 composite assimilates water and it is permitted to spread in the substrate and making it hydrophilic. Be that as it may, CMC composite film are hydrophobic in nature.

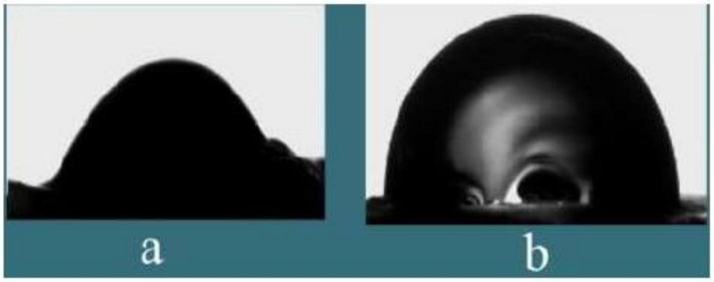

Figure 7. Contact angle measurements of a) NCC/chitosan/Fe2O3 b) CMC.

3.6. Antibacterial activity

Inhibitory impact of cellulose based film and CMC film against bacterial strains like *E*, coli, S.aures are appeared in Fig.8. The inhibitory impact was estimated dependent on clear zone encompassing. On the off chance that there is no unmistakable encompassing the film, it is accepted that there is no inhibitory zone. As far as clear encompassing zones, the film show inhibitory impact against allmicro organisms. It is obvious from the figure that CMC shows better obstruction toward both the micro organisms.

Figure 8. Anti microbial activity of (1&2) cellulose composite against E.coli and S. Aurus (3&4) CMC composite against E. coli and S. Aurus.

4. Conclusions

In this investigation cellulose isolated from peanut shell based biodegradable film were prepared, characterized and applied for the food packaging application. The prepared biodegradable polysaccharide film is attractive thinking about natural and financial angles. Expansion of polysaccharide by addition of chitosan increased the thermal stability of the films. The surface morphologies demonstrated better homogenization of the biopolymers. Surface change and expansion of iron nano particles expanded the antimicrobial activity of the film. The prepared film inhibit the growth of specific sort of microorganism like E.coli, S.aureus. Antibacterial test demonstrates that surface altered composite is better decision for the food packaging use. It tends to be inferred that the modified nano cellulose isolated from peanut shell fortified biodegradable polymer-based film can be viably utilized as packaging material to upgrade the time span of usability of food materials.

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