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Synthesis, Characterization and Biological Activities of new Nano Schiff bases Composites

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Abstract

New nano composites containing Schiff bases have been synthesized and presented in this paper. All compounds have been categorized through FT-IR and some of them by H-NMR spectroscopy. The antibacterial performance of the prepared compounds has been investigated according to the agar diffusion method. The compounds (P1, P2, C1, and C2) have shown, in general, significant inhibition against bacterial.

Keywords: Nanocomposite, Nanoalumina modification, Schiff bases and Biological Activities

1. Introduction

Widespread use of nano structured materials and metal oxide nanoparticles magnetize numerous chemistry scholars for their regulated synthesis through a new technique [1]. The biochemical surfaces adaptation has been investigated extensively for diverse centuries. Lately, there has been apposition attention in surface nano structuring due to its numerous uses in microelectronics, biomaterials and medicine. For instance, the surface can be modified for controlling interacting with cells [2]. Polymers have been mostly employed in technical textiles field. The prevalent applications of general organic polymers like nylons, polyolefin, polyurethanes and polyesters, are resulted from significant characteristics such as uncomplicated fabrication, lightweight, extraordinary process ability, robustness and reasonable cost effectiveness [3,4]. The generic class of nano composite organic/inorganic materials is progressively within the areas of many biochemical researches. Strong efforts are concentrated on achieving fitting control of the nano scale structures by advanced synthetic methods. The features of nano composite materials are based not just on the feature of their specific parents but as well on their interfacial and morphology features [5]. Polymer nano composites are featured as composites with elements based on the nanometer scale. Macromolecules and nano composites have numerous key advantages including higher

Interface area between fillers and matrix [6]. Many of the inorganic minerals have hydrophilic surfaces and are not favorable with most hydrophobic polymer matrices. The purpose of fillers surface modification is to confirm their hydrophobization with the polymer in order to make intercalation or exfoliation [7]. Chemical grafting of polymers onto inorganic particles can be gotten via covalent bonding with the hydroxyl groups on the unmodified particle surface [8, 9]. For more than hundred and fifty years, schiff bases (SBs) have still been Avery essential kind of organic compounds in chemical, medical, biochemical, and pharmaceutical areas. SBs have been given a huge range of organic activities like antimalarial, antipyretic, and antibacterial activities [10].

2. Materials and Methods

2.1 Materials

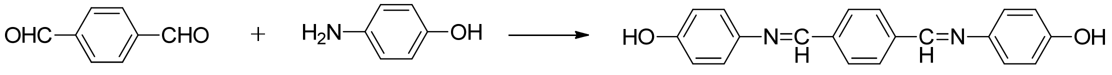
Alumina Nano (80nm), 4-Amino phenol from (BDH), Benzoyl peroxide from (MERK) Tere Phthaldehyde, P-Bromo benzaldehde from Alaf,

2.2. Techniques:

The FTIR spectra have been read by Shimadzu 8300 Fourier Transform ranging from 400-4000cm-1. H-NMR spectrums have been read via Brucker 60 MHz spectrometer.

2.3. Preparation of 4,4'-((1,4- phenylenebis (methaneylylidene)) bis (azaneylylidene)) diphenol (1): [11].

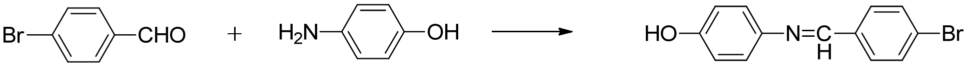
A solution of (0.01 mol), 1.34gm, of (Tere Phthaldehyde) in (20) ml of absolute ethanol, (0.02 mol),2.18 gm of (4-aminophenol) was added. Then, number of drop glacial acetic acid was added as a catalyst. The reaction mixture was heated under reflux for 4 hours. Then, this reaction mixture was cooled and the product was stirred. Lastly, it was filtered and purified by recrystallization based on hot water with m.p (264-266) ◦C and yield 79.5%



1

2.4. Preparation of 4-((4-bromobenzylidene) amino) phenol (2): [12].

Compound 2 was prepared by the same preparation procedure of 1 but with using 0.01mol, 1.85 g of 4-bromobenzaldehyde with 0.01mol, 1.09 gm of 4-aminophenol with m.p (210-211) and yield 82.65%.



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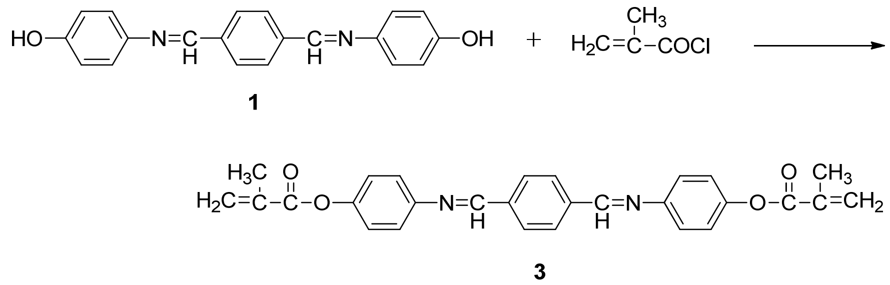
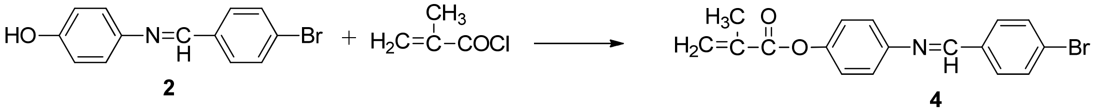
2.5. Preparation of prop-1-en-2-yl 4-((4-(((4 (methacryloyloxy) phenyl) imino) methyl) benzylidene) amino) benzoate (3):[13].

The 1.58g, 0.01 mol of compound (1) was dissolved in 15 mL tetrahydrofurane (THF) with 2ml of trietheylamine (TE3N). Afterward, 2.09 g, 0.02 mol of methacryloyl chloride was inserted drop wise with stirring in (0°C) for 5 hours. To the cold mixture, 50 mL of ice water was inserted. The precipitate was filtered and washed by water several time. Then, sodium bicarbonate was dried under room temperature with m.p (110-112) and yield of 83%.



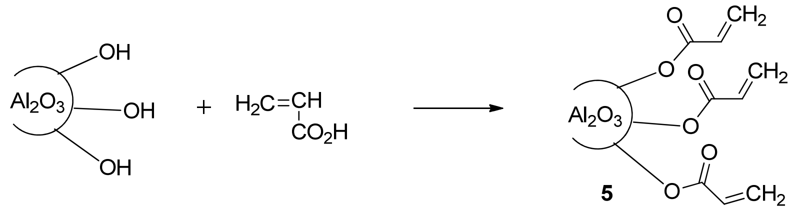
2.6. Preparation of 4-((4-bromobenzylidene) amino) pheny methacrylate (4): [13].

Compound 4 was prepared by the same preparation procedure of compound 3 but using 0.01mol, 2.76 of compound 2 with 0.01mol, .045 gm of methacryloyl chloride.m.p (99-101),yield 80%.



2.7. Preparation of Nano alumina –Graft AcrylicAcid Monomer (5): [14].

About 1.0 g of nano alumina in flask with 20 ml toluene was stirred. At that time, 3.0 g of acrylic acid monomer was added drop wise. The reacted mixture was left for stirring process for (8) hour. Subsequently, the product was filtered and washed initially with 15 ml distilled water and secondly with 15 ml acetone, and dried then under room temperature.



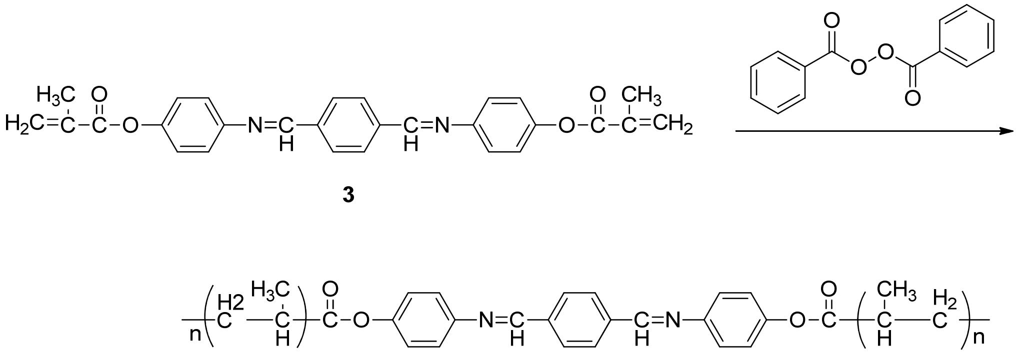
2.8. Preparation of Polymers (P_1, P_2): [15].

2.8.1Preparation of P1

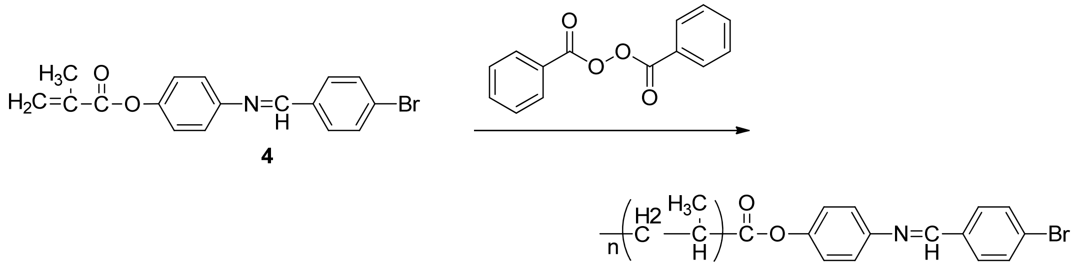
In a two-neck round-bottom flask, (25) ml of toluene as a solvent was positioned under nitrogen atmosphere in controlled water bath at (80°C). After 3 minutes, 0.15 g of benzoyl peroxide was inserted. Then, 3 g of compound 3 was added, and left for stirring process for 5 hours. The product was filtered and washed primarily with 10 ml toluene and then with 10 ml acetone, dried under room temperature.

2.8.2Preparation of P2

Polymer P2 was prepared based on the same method of polymer P1, using compound 4.



P1 (6)



P2 (6)

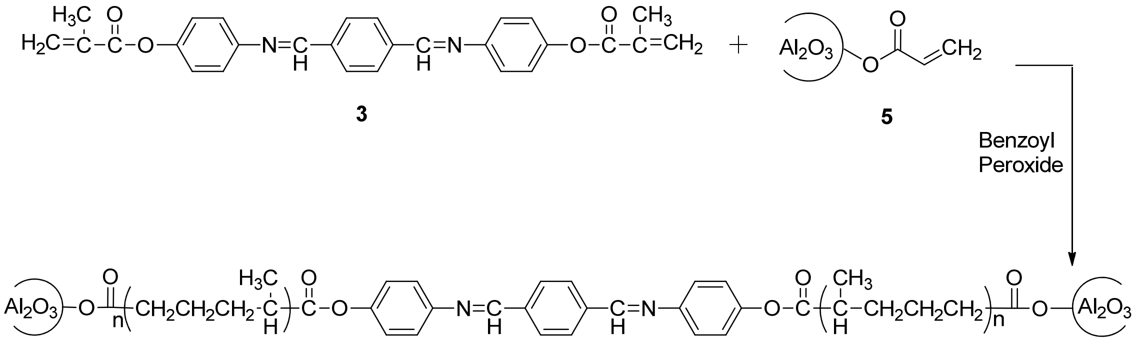
2.9. Preparation of Nanocomposites (C_1, C_2): [15]

2.9.1 Preparation of C1

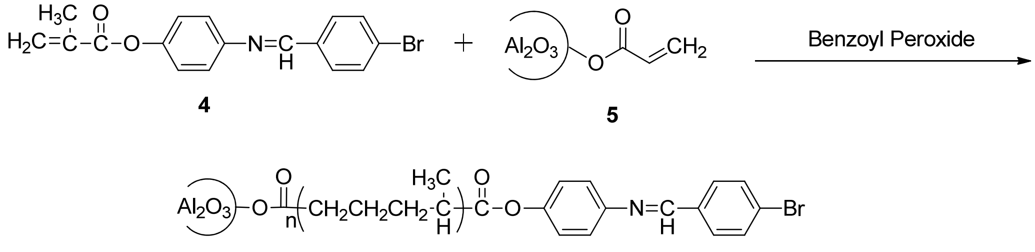
Nanocomposite C1 was prepared by the same process of P1 but after inserting benzoyl peroxide. About 2.5 g nano alumina grafted acrylic acid and then 2.5 g of compound 3 were sequentially added.

2.9.2 preparation of C2

This Nanocomposite was prepared by the same sort of C1 but with compound 4.



C1 (6)



4 5

C2 (6)

2.10. Biological Activity

The antibacterial- activity of the prepared compounds (P_1, P_2, C_1, and C_2) was performed in relation to the agar diffusion method. The prepared compounds were tested against *bacillus ceru, bacillus sabtillus, staph*. Each compound was dissolved in 2ml DMSO in concentration (0.024 M). The plates were then incubated at 37ºC and examined after 24 hrs.

3. Results and Discussion

The structures of all synthesized compounds were confirmed by using FT-IR spectral. It was a good evidence corresponding to the structure of suggested compounds and some of the compounds by H-NMR.

3.1. Characterization of Schiff-baes Compounds (1, 2).

The compound (1) was prepared by reacting Tere Phthaldehyde with 4-amino phenol in absolute ethanol. **Figure 1.** Depicts browed peak at (3417) cm-1 that is related to the (O-H), stretching. An observed band at 1620 cm^{-1} is assigned to C=N isomethane, and showed disappeared band of primary (NH_2) and carbonyl of aldehyde. The compound (2) has equipped by reacting 4-bromobenzaldehyde with 4-amino phenol in absolute ethanol. **Figure 2.** Depicts the following absorption bands:
O-H stretching is at (3105) cm^{-1}. It as well shows a peak at (1621) cm^{-1} due to the C=N and also disappeared band of primary (NH_2) and carbonyl of aldehyde.

3.2Characterazation of Esters Compounds (3, 4).

Compounds (3, 4) were categorized through FT-IR and H-NMR spectral tools. The compound (3) was prepared by reacting compound (1) with methacryloyl chloride in THF. Based on FT-IR, **Figure 3.** Illustrates the strong peak at (1743) cm^{-1} that is related with the C=O stretching. Stretching band of (C=N) showed absorbance at 1618cm^{-1} with disappeared band of O-H. The H-NMR spectrum for compound 3 showed signals at d = 1.25 ppm for 3 protons of (CH_3) groups signals seemed at d =6.82 ppm for two protons of (CH_2) groups with multiple signals in the region with d =7.3-8.4 for aromatic protons. The signal at d=9.99 ppm is for one proton of C=N as in **Figure 4.** The compound 4 xprimed by reacting compound (2) with methacryloyl chloride in THF. **Figure 5.** X illustrated disappeared band of O-H, band at approximately 1737 cm-1 related with stretching vibration (C=O) of ester. The stretching band of (C=N) showed an absorbance at 1612cm-1. It also showed appearance features bands at (2916-2850) $cm\text{-}^{1}$ of Aliphatic (CH_3). The H-NMR spectrum showed signals at d = 1.19 ppm for three protons of (CH_3) group, multiple signals in the region of d =6.5-7.5 for aromatic protons. The signal at d=9.49 ppm was for one proton of C=N as explained by **Figure 6.**

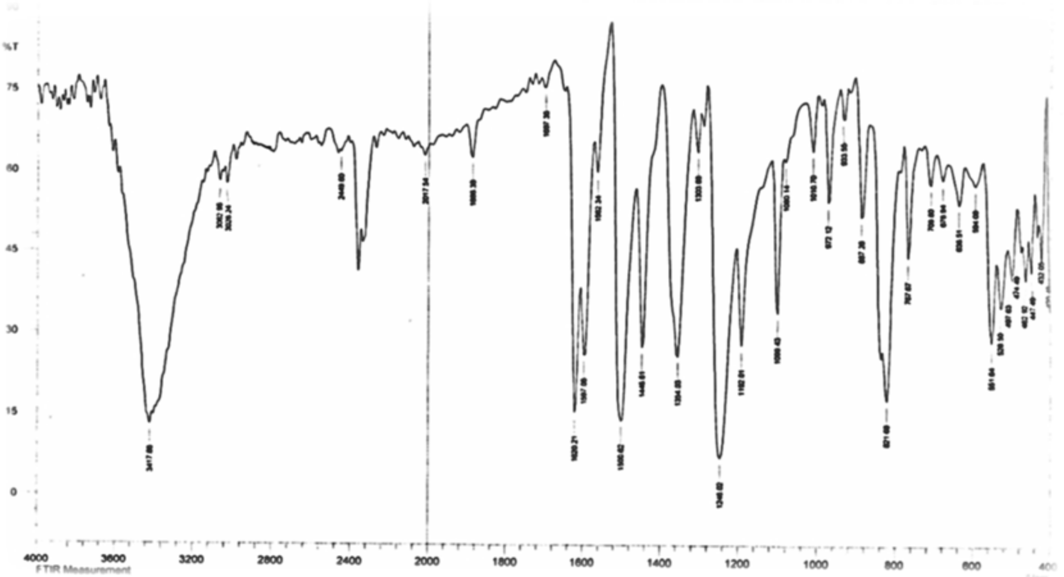


Figure 1. FT-IR of compound 1.

3.3Characterazation of Nanoalumaina-graft Acrylic Acid Monomer.

Nanoalumina-graft acrylic acid monomer was organized by grafted nanoalumina with acrylic acid. The structure of this compound was investigated through FT-IR spectroscopy. By FT-IR, **Figure 7.** Depicts the following absorption bands C=O stretching at 1751cm-1. Also, it shows the peak at (3487) cm^{-1} as a result of the O-H.

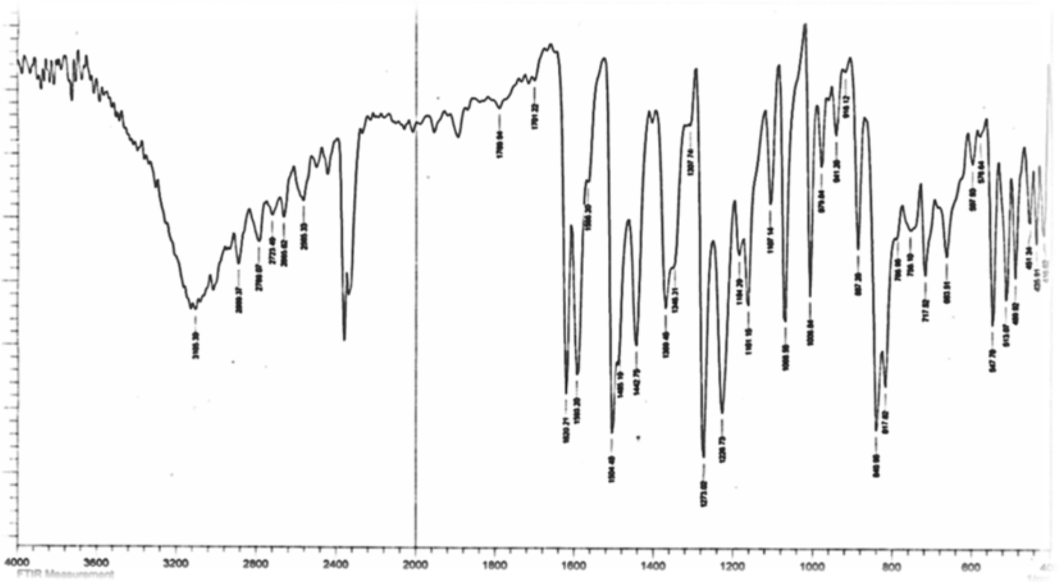


Figure 2. FT-IR of compound 2.

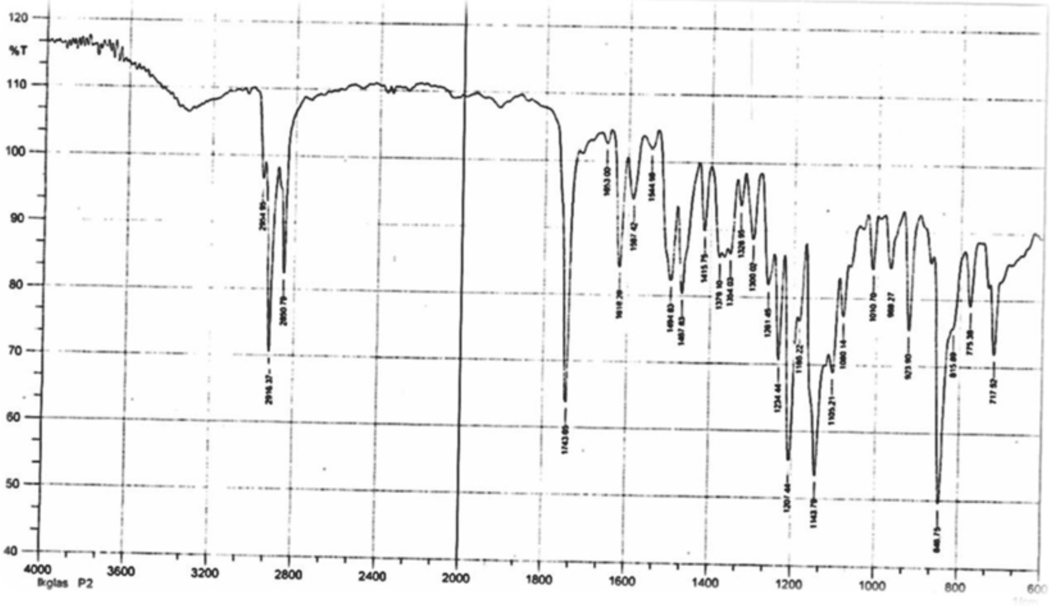


Figure 3. FT-IR of compound 3.

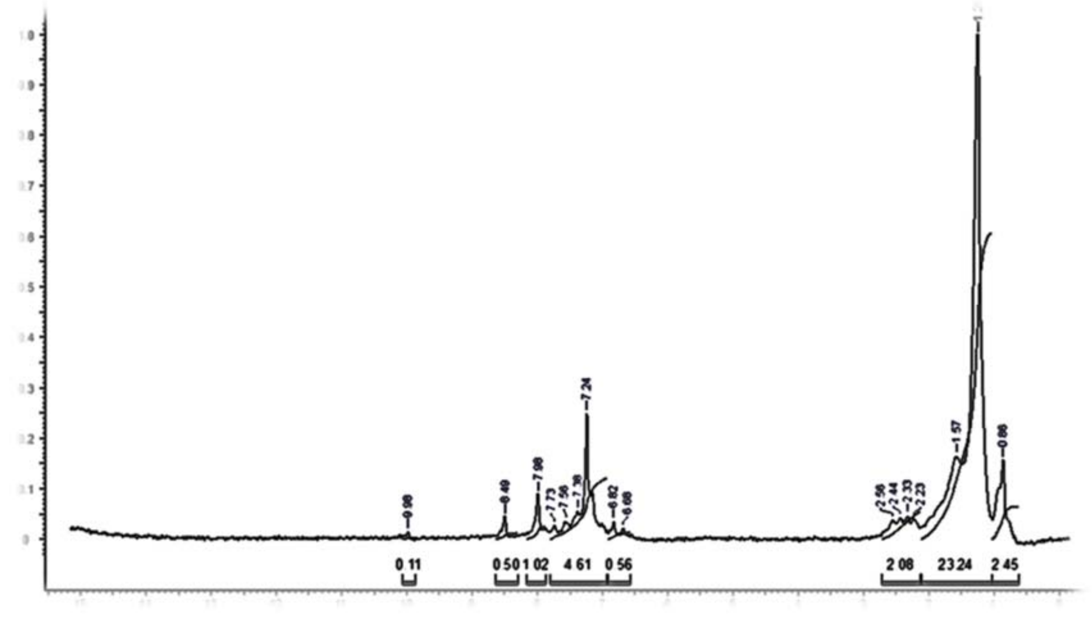


Figure 4. H-NMR of compound 3.

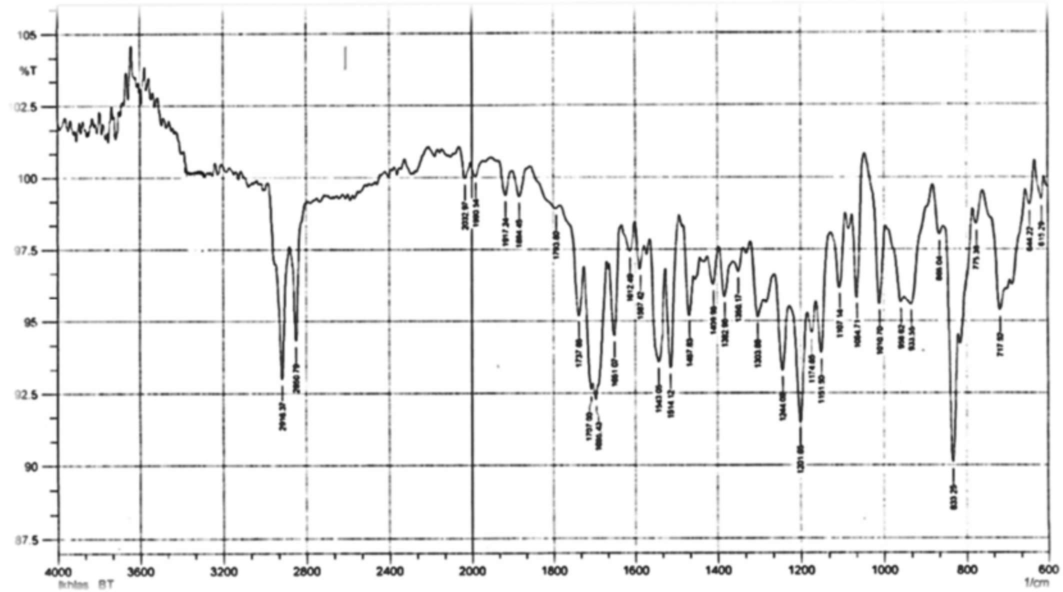


Figure 5. FT-IR of compound 4.

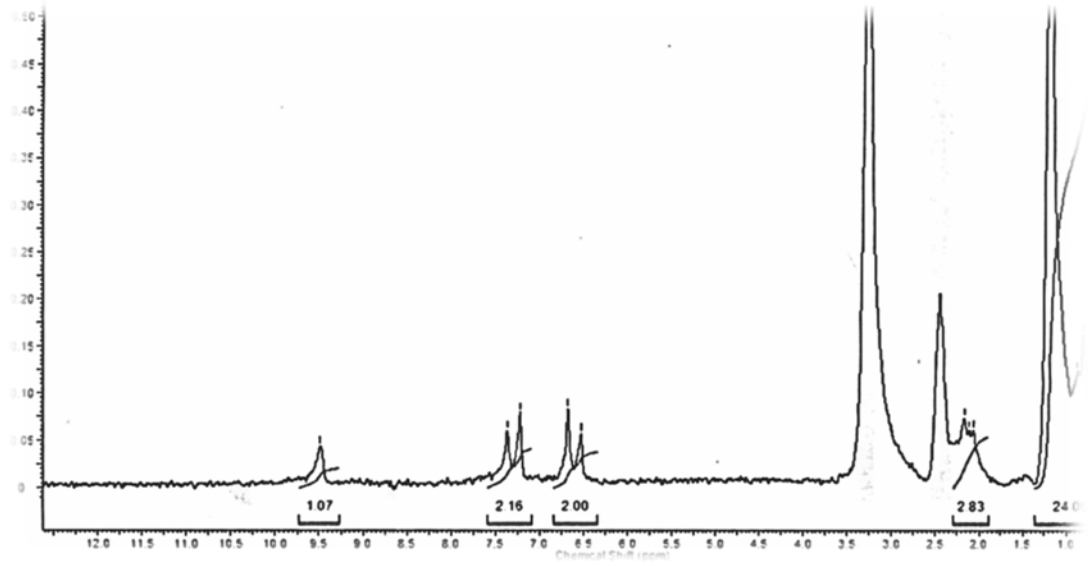


Figure 6. H-NMR of company 4.

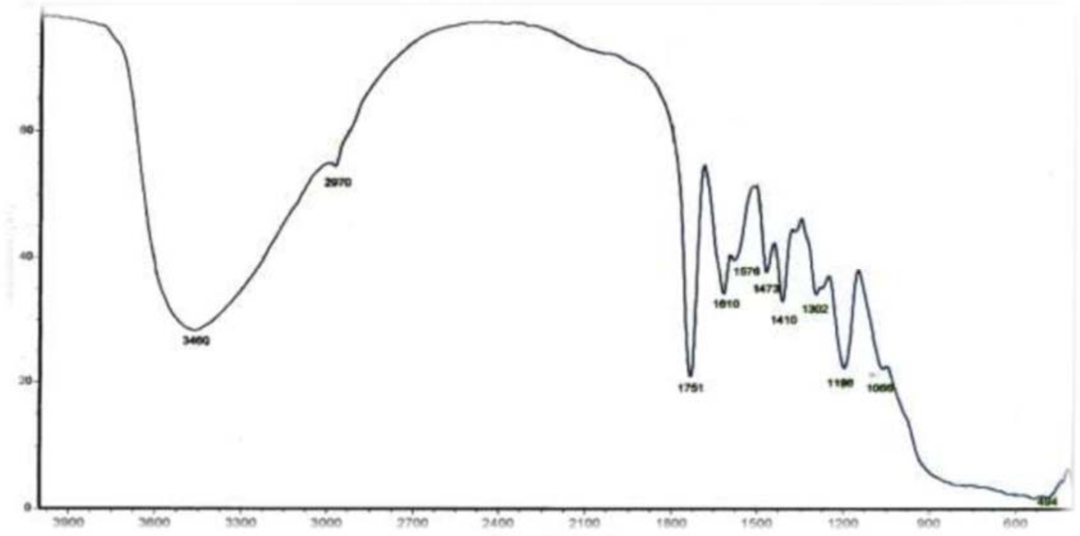


Figure 7. FT-IR of alumina graft acrylic acid monomer.

3.4. Charchteraztion of Polymer (P1, P2)

The polymers were prepared by free radical polymerization. Those polymers were considered by FT-IR spectroscopy. The characteristic absorption bands of the polymer (P1) as in **Figure 8.** Showed strong peaks at (2916- 2850) cm-1 of absorption. Alkyl groups (C-H) and the FT-IR spectrum also showed disappearance the peak of (C=C) group which verified the formation of the polymer. **Figure 9.** For polymer (P2) showed peaks at (2916- 2848) cm-1 of absorption Alkyl groups (C-H). FT-IR as wellx depicted disappearance of a peak of (C=C) group.

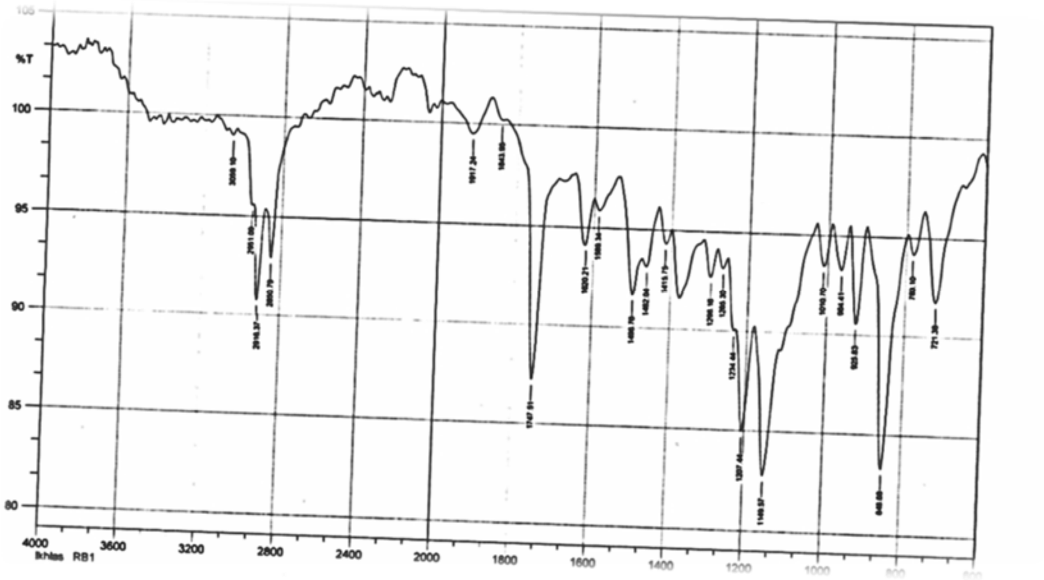


Figure 8. FT-IR of polymer P1.

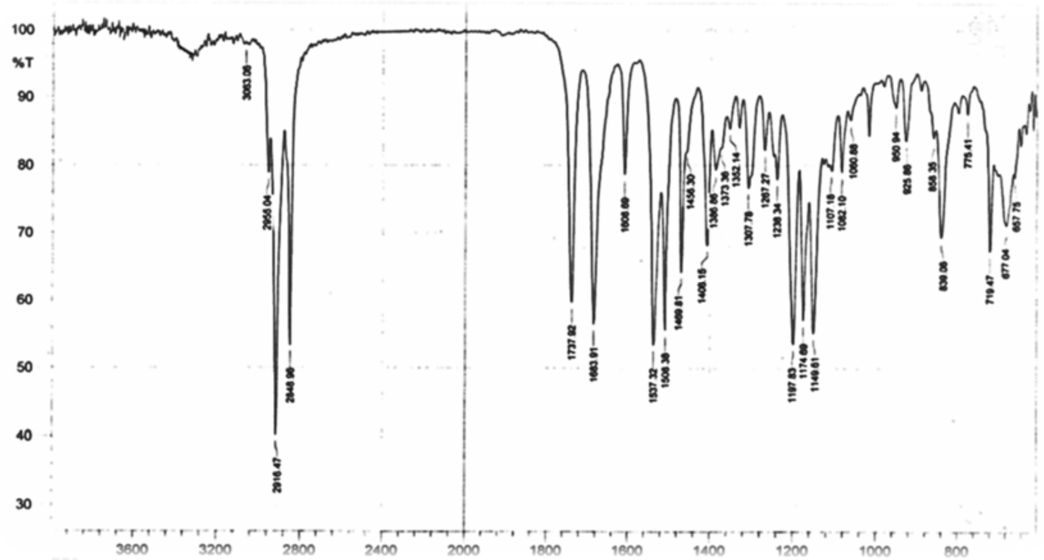


Figure 9. FT-IR of polymer P2.

3.5. Charchteraztion of Nanocomposite

The nanocomposite was prepared by polymerization of nanoalumina grafted – acrylic acid once with compound 3 once more with compound 4. By FT-IR spectrum, **Figure 10.** Of nanocomposite C1 showed the appearance of x stretching bands of alkyl groups (C-H) at (2927-2850). On the other hand, FT-IR disappearance band of (C=C) group. The FT-IR spectrum of nanocomposite C2 in **Figure 11.** Depicted strong band in region between (2916-2848) due to absorption of alkyl groups (C-H) and also disappearance band of (C=C) group.

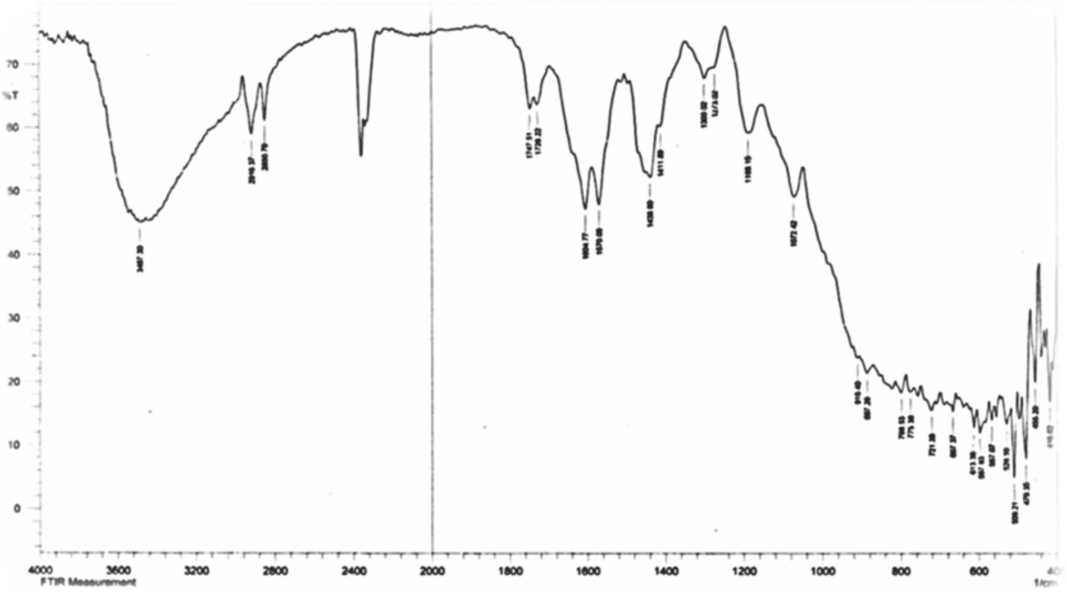


Figure 10. FT-IR of nanocomposite C1.

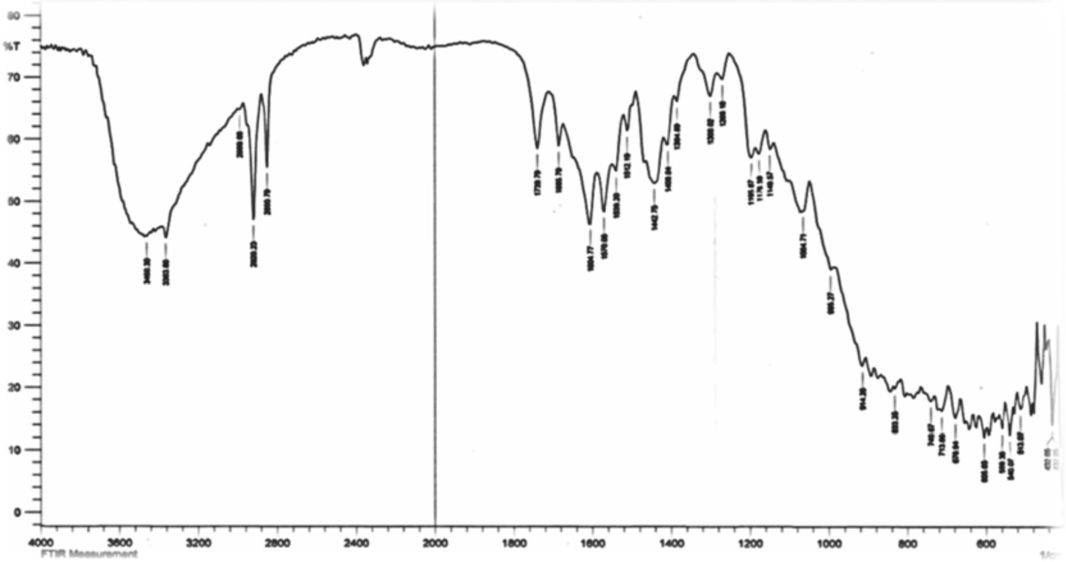


Figure 11. FT-IR of nanocomposite C2.

3.6 Biological activity Results.

The resulting data of biological activity were listed in **Table 1.** All compounds (P1, P2, C1, and C2) were given inhibition in contradiction of bacteria (*b. staph , b.cerus*). But with (*b. sabtillus*), there was no any inhibition.

Table 1. Biological Activity Result.

No	Sample	b.staph	b.cerus	b.sabtillus
1	P1	17mm	17mm	___
2	C1	18mm	14mm	___
3	P2	14mm	18mm	___
4	C2	15mm	17mm	___
5	stander	14mm	27mm	16mm

4. Conclusions

In this article, new nanocomposites were created, and their biological activities were investigated regarding their structure. The chemical structures of these nanocomposites and their polymers were examined by FT-IR spectroscopy. The results were in agreement with the considered molecular structure.

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