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PREPARATION, CHARACTERIZATION AND ANTIMICROBIAL ACTIVITY OF POLYVINYL ALCOHOL/ POLYVINYL PYRROLIDONE/ CHITOSAN NANO COMPOSITE

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ABSTRACT : Ternary polymer blend of chitosan/poly vinyl alcohol/ poly vinyl pyrrolidone was prepared by solution casting method, nanocomposite was prepared by sonication method with nano Ag and Zn. All prepared compounds have been characterized by FT-IR, SEM, DSC, as well as Biological activity. Antimicrobialactivity related to prepared blendsand Nanocomposites against six types of bacteria namely, *Staphylococcus aureas*, *E. faecalis*, *S.typhi*, *P. aeruginosa, Bacillus subtilis*, *Escherichia coli* and *C. albicans* fungal were examined and evaluated. The results reveal that the prepared polymer blends and nanocompositeshave good antimicrobial activity against all kinds of microbials.

Key words : Chitosan, PVA, PVP, polymer blend, nanocomposite, antimicrobial polymer.

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INTRODUCTION

Bionanocomposites are made up of a biopolymer matrix (continuous phase) and a reinforcing agent (dispersed phase) made up of particles with sizes ranging from 1 to 100 nm (Joseph *et al*, 2020). Most of the biopolymers are inherently weak in physical, mechanical and thermal attributes compared to widely-used petro-based polymer (Garavand *et al*, 2020). Nanobiocomposites possess a couple of advantages over the biocomposites and even composites; in turn, loading nanofillers into the biocomposites could improve their thermal, structural, mechanical and barrier attributes in a safe way with widespread range of uses (Wei *et al*, 2020; Rezkita *et al*, 2020).

Chitosan is a polycationic linear polysaccharide produced from chitin that is naturally polycationic. Chitosan's poor solubility in neutral and alkaline solutions restricts its use. Chemical modification of composites or hydrogels, on the other hand, confers new functional characteristics for a variety of applications. Because of its low allergenicity, biodegradability, biocompatibility andnon-toxicity, chitosan is regarded as a versatile biomaterial (Kumar *et al*, 2020; Cheung *et al*, 2015). Chitosan's unique properties enable it to be utilized in a wide range of applications. Other biological characteristics of chitosan have been identified, including anticancer, antibacterial, and antioxidant capabilities (Aranaz *et al*, 2009). Chitosan has many approved biological properties. It is available in nature and can be fabricate into many forms like film, fiber, bead and powder (Jou *et al*, 2011). Despite the fact that Chitosan is not predominantly employed as an antibacterial agent, its usage as a component in food and pharmaceutical formulations has recently increased, and the pharmacological actions of this versatile carbohydrate have begun to emerge. Understanding the many variables that influence its antimicrobial action, on the other hand, has become a critical problem for improved chitosan formulation use and optimization (Raafat *et al*, 2009; Martins *et al*, 2014; Kassem *et al*, 2019). Chitosan has been recognized as a potential antifungalsubstance (Qin *et al*, 2020).

PVA (polyvinyl alcohol) is a non-toxic, water-soluble synthetic polymer with strong physical and chemical characteristics as well as the capacity to create films (Parida *et al*, 2011). Because of their permeability, biocompatibility and biodegradability, chemically crosslinked PVA hydrogels are gaining popularity in biomedical and biochemical applications (Aytimur *et al*,

2013). PVA is a kind of versatile polymer and contains high mechanical applications (Arefian *et al*, 2020).

PVP (polyvinyl pyrrolidone) is a synthetic polymer with non-toxic, bio-inert and hydrophilic characteristics, making it a promising option for drug delivery applications in the pharmaceutical industry (Kumar, 2014). PVP is a binder utilized in many pharmaceutical tablets and PVP complexes are used in a variety of goods such as solutions, ointment, pessaries, liquid soaps and surgical scrubs (Karpuraranjith *et al*, 2017). In polar solvents such as alcohol, water, and others, PVP is highly soluble. It also has a high glass transition temperature (Tg) and good environmental, thermal and mechanical stability. Another advantage of PVP is that it forms a thermally cross-connected polymer chain, which gives it exceptional thermal stability and great mechanical strength in mix composites. Poly(vinyl pyrrolidone) (PVP) was chosen as another polymer to construct polymer blends because of its outstanding properties (Sundaramahalingam *et al*, 2019).

MATERIALS AND METHODS

Polymer blend preparation

The polymer blend was prepared by solvent casting method. Chitosan solution was made by dissolving it in a 2 percent aqueous acetic acid solution and stirring it at room temperature. PVA and PVP were dissolved in hot water to form 5 wt% polymer solutions.

Preparation of Ch/PVA/PVP-Ag, Zn nanocomposites

A 100 mg dried PVA/PVP/Ch mix was put in 50 mL of Ag and Zn solution at a concentration of 250 mg/L and sonicated for 1.5 hours to electrostatically bind Ag and Zn nano metals in the blend matrix.

RESULTS AND DISCUSSION

FT-IR analysis for polymers and polymer blend

From Fig. 1 for PVA, the bands at about3369 and 1649cm^{-1} are assigned to stretching and bending vibration for hydroxyl group (Li *et al*, 2000). The band 2953cm^{-1} corresponding to asymmetric stretching vibration for (CH_2) group. The band about1151cm^{-1} for theacetyl groups (C–O) stretching vibration on the PVA backbone (Abdelaziz *et al*, 2007; Laot *et al*, 1999). The peak at 3392 cm^{-1} in the FTIR spectrum of PVP (Fig. 2) shows O-H stretching. The peaks at 2923 and 1623 cm^{-1}, respectively, showed the presence of asymmetric CH_2 and C-O stretching. At 1457 cm^{-1} and 1383 cm^{-1}, respectively, C-H bending and CH_2 wagging were detected (Rahma *et al*, 2016). The FTIR spectra for pure Chitosan (Fig. 3) can be assigned: Broad band at 3389cm^{-1} for stretching vibrations of (N–H and O–H) groups, 2953cm^{-1} (CH_3 symmetric stretch), 1650cm^{-1} (C=O stretching vibration), 1458cm^{-1} (C–N stretching vibration), 1377cm^{-1} (CH_3 bending vibration), 1153cm^{-1} (C–O–C bending vibration). For the prepared polymer blend. Fig. 4 showed a broad band around 3417cm^{-1} attributed to stretching vibration of hydroxylgroup (–OH) of PVA and the secondary amide (–NH) of chitosan. The band at around 1046cm^{-1} indicates the presence of (OH) hydroxyl group with polymeric association and (–NH) a secondary amide. Also band appeared at 1452cm^{-1} assigned to pyridine ring (C=N).

Scanning electron microscope studies (SEM)

The SEM micrograph for PVA/ PVP/Ch polymer blend and nanocomposite loaded with silver and zinc nanoparticles are represented in Figs. 5-7. The surface seems to be porous with some inclusions. Nanoparticles with an average size of 69nm for silver and 72nm for zinc particles are found in a homogeneous distribution over the matrix's surface.

Thermal analysis

The thermo gravimetric (DSCD TGA) for PVA/ PVP/Chitosan polymer blend and its nano composite has been measured in temperature, which ranges between 25°C and 600°C with a constant rate which is equal to 10°CD min^{-1}.

TGA curve of PVA/PVP/Chitosan polymer blend (B1), Fig. 8 illustrated four stages of a sequence mass lose, the first stage with mass lose (-8.935%) of volatile compounds. The second-step with weight loss approximately (-24.85%), the third stage with weight loss of approximately (-55.47%), the fourth stage with weight loss of approximately (-6.528%) for the chain decomposition. DSC curve in the Fig. 9 for polymer blend showed a Tg of (118.19°C). Peak at (446.81°C) regarding to the polymer melting Tm.

The TGA curve of PVA/PVP/Chitosan -Ag nanocomposite (NC-Ag), Fig. 9 illustrated four stages of a sequence mass lose, the first stage with mass lose (-10.18%) of volatile compounds. The second stage with weight loss approximately (-23.71%), the third stage with weight loss of approximately (-60.29%), the fourthstage with weight loss of approximately (-4.151%) for the chain decomposition. DSC curve in the Fig. 9 for polymer nanocomposite showed a Tg of (98.04°C), peak regarding to the Crystalline temperature point Tc at (351.98°C). Peak at (444.63°C) regarding to the polymer melting Tm.

The TGA curve of PVA/PVP/Chitosan -Zn nanocomposite (NC-Zn), Fig. 10 illustrated four stages of a sequence mass lose, the first stage with mass lose (-

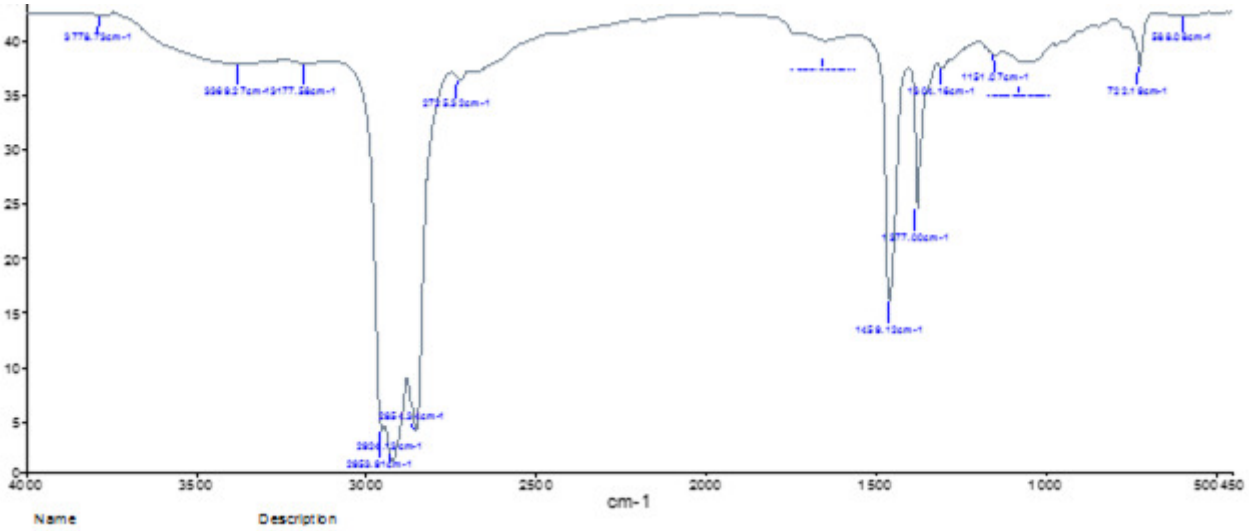

Fig. 1 : FT-IR spectrum PVA.

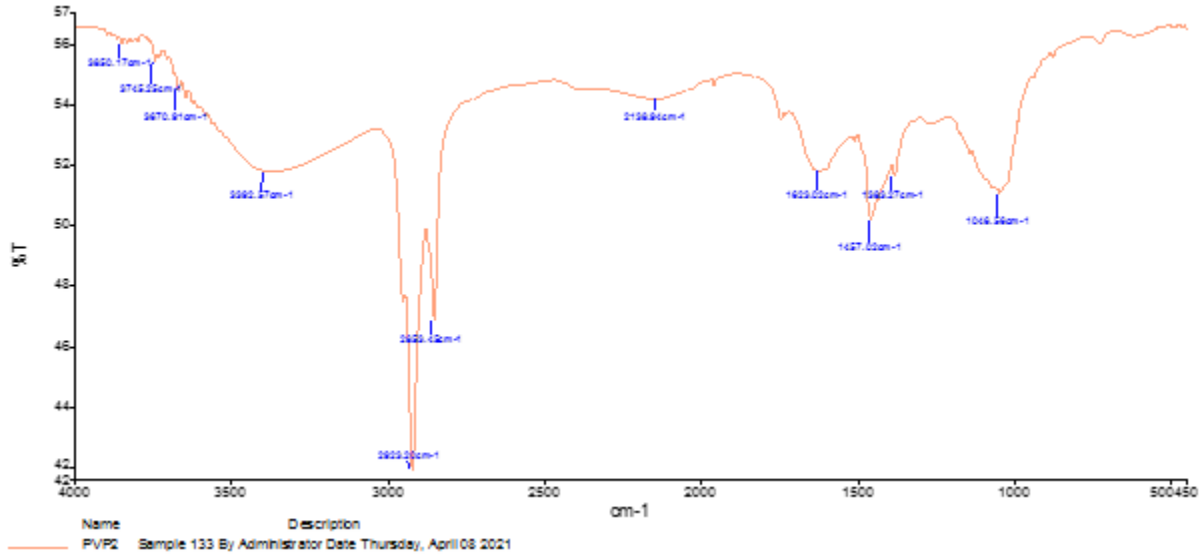

Fig. 2 : FT-IR spectrum PVP.

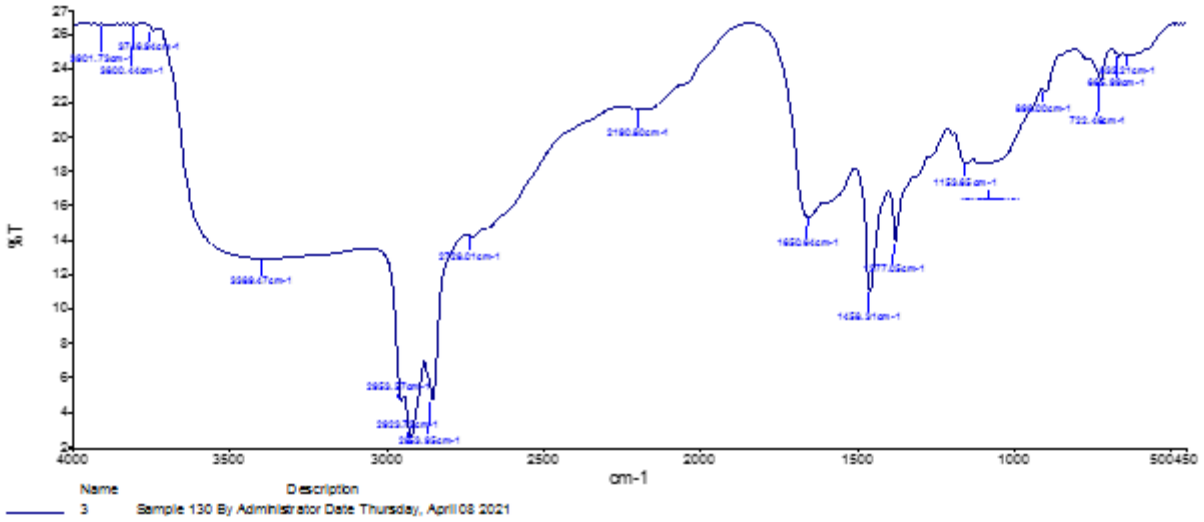

Fig. 3 : FT-IR spectrum Chitosan.

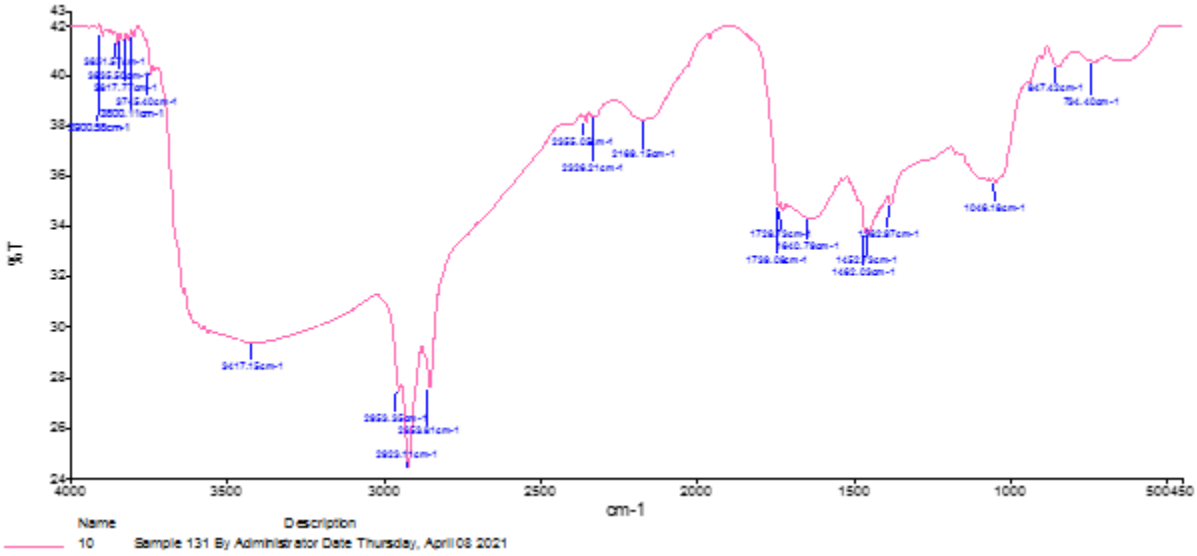

Fig. 4 : FT-IR spectrum PVA/PVP/Ch Blend.

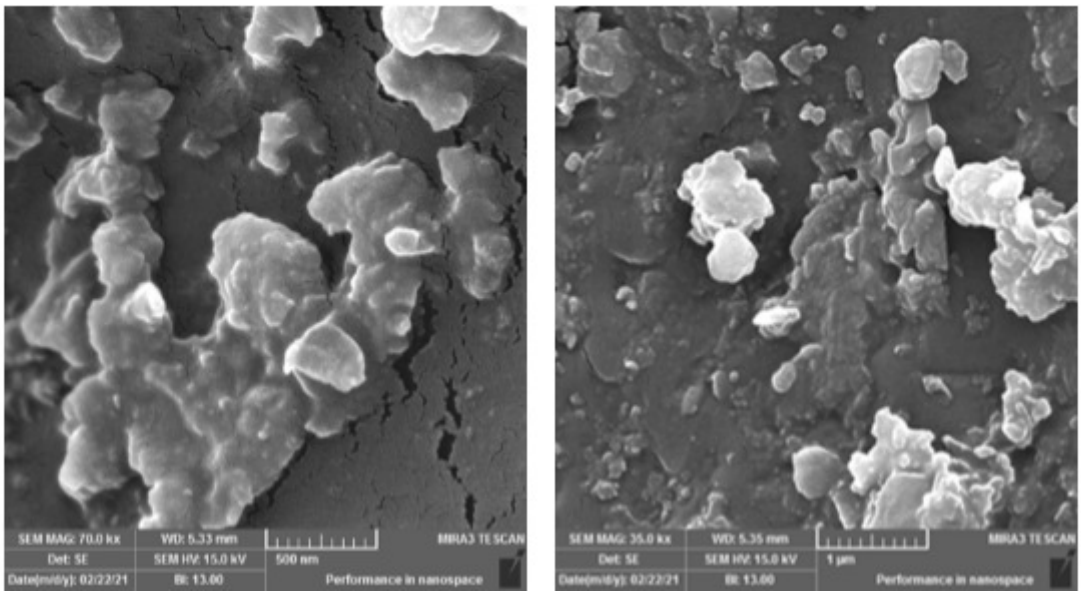

Fig. 5 : The SEM Images of blend B1.

10.61%) of volatile compounds. The second stage with weight loss approximately (-23.81%), the third stage with weight loss of approximately (-56.87%), the fourthstage with weight loss of approximately (-5.174%) for the chain decomposition. DSC curve in the Fig. 10 for polymer nanocomposite showed a Tg of (142.19°C), peak regarding to the Crystalline temperature point Tc at (311.84°C). Peak at (445.11°C) regarding to the polymer melting Tm.

Biological activity

The biological activity of the polymer blend of PVA/PVP/Chitosan Ag and Zn nano composite were tested against six types of pathogenic bacteria using Diffusion inhibition method.The results of antimicrobial activity are represented in Table 1. When compared to Ag and Zn nanocomposites, the ternary mix (PVA/PVP/Chitosan) that was employed as a control matrix demonstrated minimal antibacterial activity. It is clear from Table 1 that all tested compounds exhibit good antimicrobial activities.

For Ag nanocomposite the silver exhibits antibacterial property, which leads to biomedical applications.Silver's antibacterial action is based on Ag^{+}, which binds tightly to electron donor groups in microbials cell wall such as sulfur, oxygen, or nitrogen. Silver ions work by displacing other important metal ions like Ca^{2+} and Zn^{2+} (Boomi *et al*, 2013). Silver nanoparticles have complex impacts on bacterial cells (Kim *et al*, 2011). However, the impact of silver nanoparticles on bacterial cells is mediated by a variety of ways (Prabhu *et al*, 2012). The following are some of the mechanisms that have been summarized and presented: I the capacity of silver nanoparticles to bind

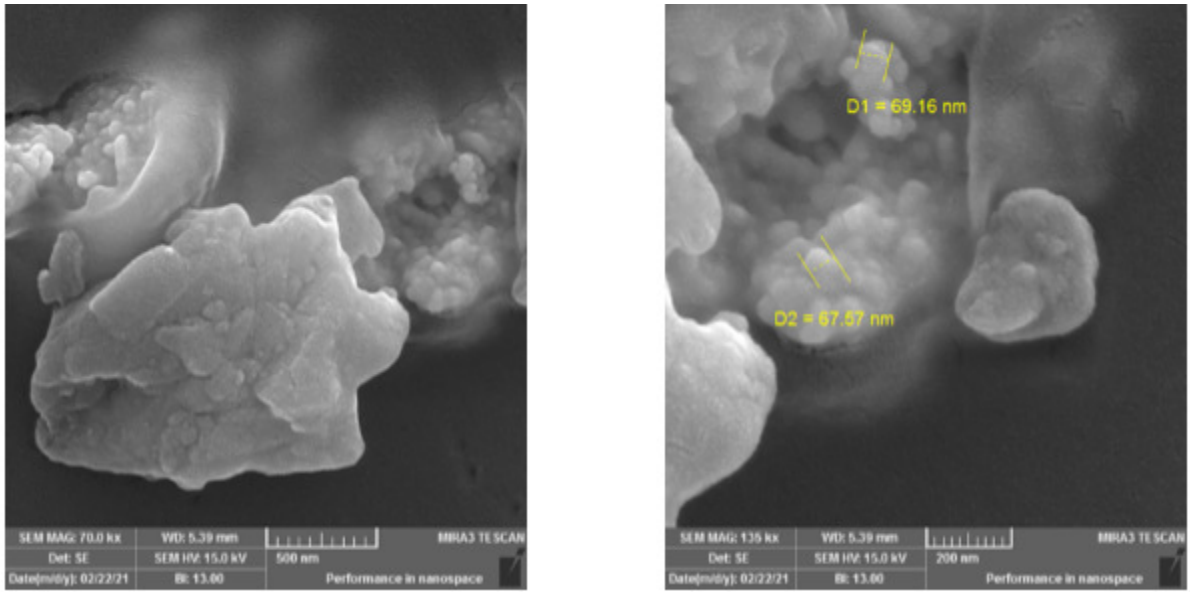

Fig. 6 : The SEM Images of Nanocomposite B1-Ag.

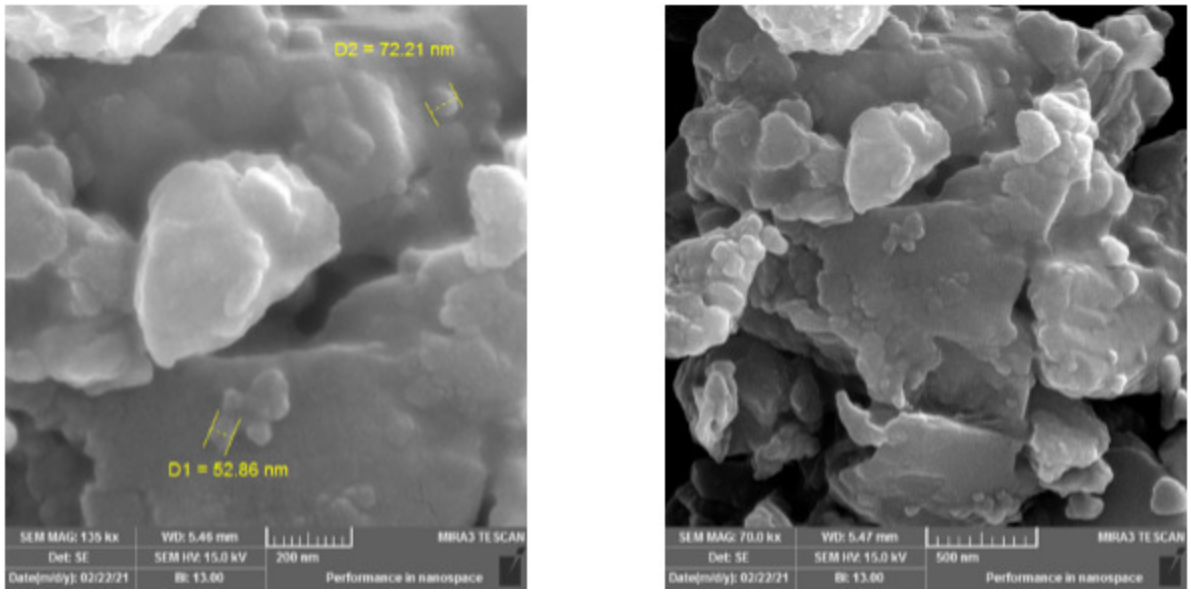

Fig. 7 : The SEM Image of Nanocomposite B1-Zn.

Table 1 : Antimicrobial activity of Polymer blend and Ag, Zn-nanocomposite on the bacterial and fungal isolates by well diffusion test.

Test	*C. albicans* Inhibition zone(mm)	*S.aureus* Inhibition zone (mm)	*E.faecalis* Inhibition zone (mm)	*E.coli* Inhibition zone (mm)	*S. typhi* Inhibition zone (mm)	*P. aeruginosa* Inhibition zone (mm)	*Bacillus cereus* Inhibition zone(mm)
B1	12	11	12	14	9	-	-
B2	13	9	11	12	11	-	-
B3	11	-	12	17	10	-	-
B1- Ag	22	21	18	26	21	18	22
B2- Ag	18	15	17	22	18	16	21
B3- Ag	15	13	15	19	16	11	20
B1- Zn	21	19	18	12	11	12	16
B2 - Zn	22	16	23	13	10	10	15
B3 - Zn	19	16	18	12	13	12	15

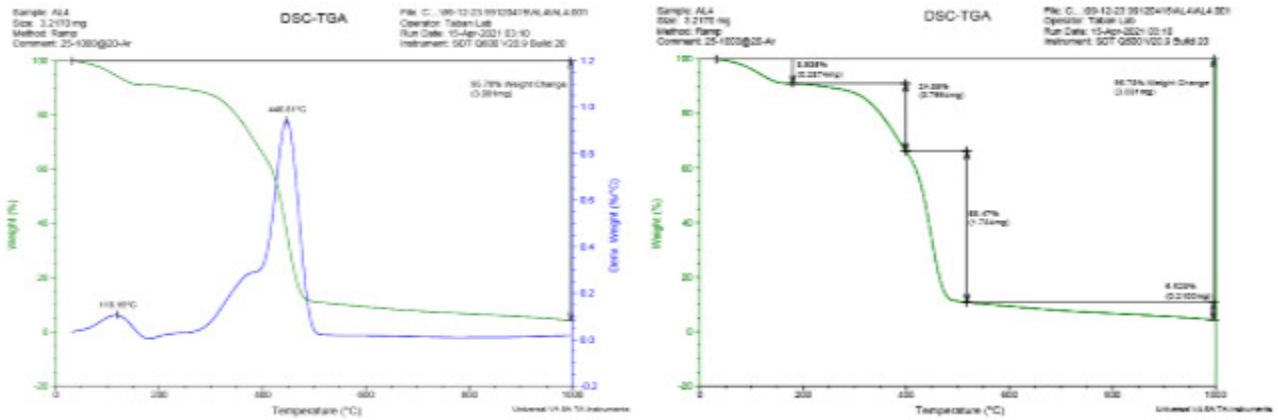

Fig. 8 : Thermal analysis of blend B1.

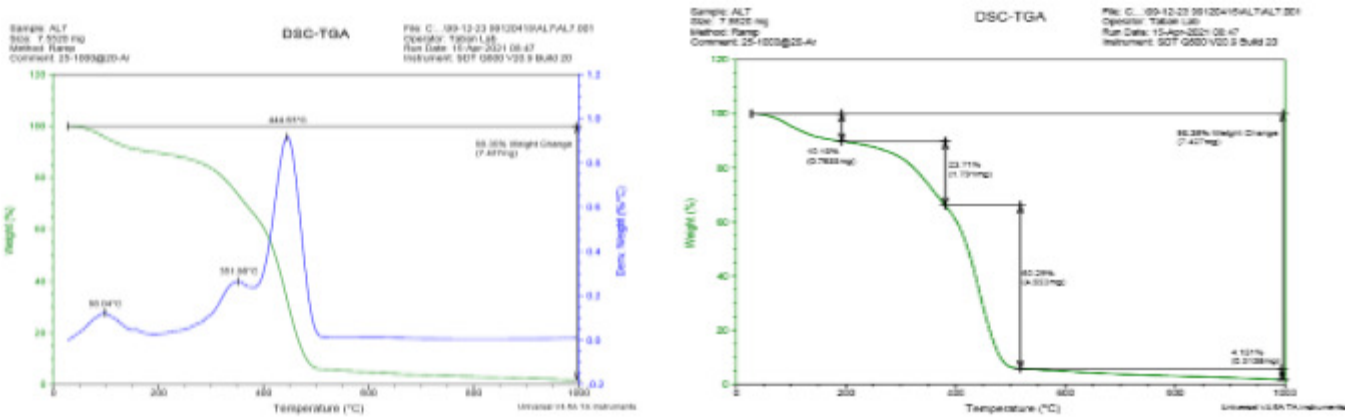

Fig. 9 : Thermal analysis of Nanocomposite B1Ag.

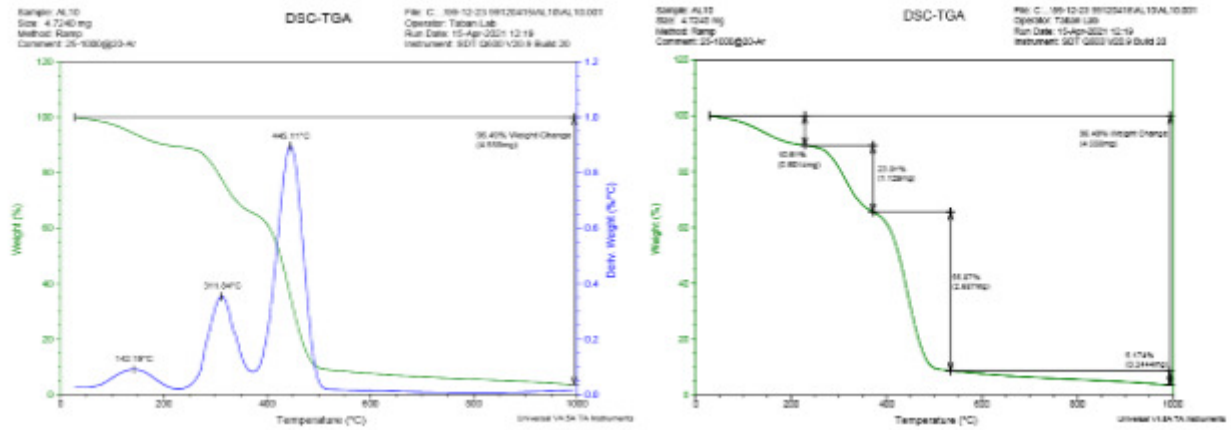

Fig. 10 : Thermal analysis of Nanocomposite B1Zn.

to and enter the bacterial cell wall (Sondi *et al*, 2004), (ii) the production of free radicals by silver nanoparticles, which may damage and porous the cell membrane (Danilcauk *et al*, 2006), (iii) nanoparticles can release silver ions, which can bind and inactivate the thiol groups of several important enzymes (Brian Y Feng *et al*, 2008), and (iv) nanoparticles can alter signal transduction in bacteria, preventing bacteria from growing (Shrivastava *et al*, 2007).

For Zn nanocomposite several mechanisms have described Zn antimicrobial activity, including cell damage through their interaction with the microorganisms and the reactive oxygen species (ROS) formation by the activation of this metal (Ghaffari-Moghaddam *et al*, 2014; Dicastillo *et al*, 2020).

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