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FUSION OF ZNO NANO/MINT EXTRACT LOADED PVP/PVA COMPOSITE BLEND: CHARACTERIZATION AND ANTIMICROBIAL ACTIVITY

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Keywords:

PVA, PVP, ZnO-NPs, SEM, TGA, FTIR, XRD, Antibacterial activity

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ABSTRACT: ZnO is used as a nanofiller in various concentrations to create flexible self-standing films of PVP-PVA mix composites. This work presents the results of structural, compositional, morphological, and optical analyses carried out using X-ray diffraction (XRD), Fourier Transform Infra-Red spectroscopy (FTIR), scanning electron microscopy (SEM), Thermo gravimetric analysis (TGA), and ultraviolet-visible spectroscopy (UV-vis). The XRD data show that ZnO nanoparticles are produced in the polymeric matrix with hexagonal phase. ZnO nanofiller is dispersed throughout the polymer matrix, as seen in SEM pictures. UV-vis spectra show that the nanocomposite films' wavelength absorption peak is between 235 and 370 nm. The blue shift may be seen with a drop in the nano filler's concentration.

INTRODUCTION:

Polymeric Blend: The most important property of polymer composites of two or even more polymers is their phase behavior. Low molecular weight solutions and polymer blends may exhibit varying degrees of extreme mixing or miscibility between the two (eg partial miscibility). Most important factor in producing miscibility of low-molecular-weight materials is the contribution of combinatorial entropy, which is significantly more than that of high-molecular-weight polymers. This work explains why solvent-solvent mixtures yield a much larger range of solubility than polymer and solvent combinations.

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The group of miscible blends, including polymer-polymer blends, is much more limited it has been [1].

Polyvinylpyrrolidone $C_{10}H_{15}NO_3$: The versatile properties of Poly (Vinyl Pyrrolidone, or PVP), including its high polarity, solubility in water and polar solvents, hardness, gloss, transparency, hygroscopicity, adhesive and cohesive properties, high degree of compatibility, and ability to be stored under normal conditions without degrading. Other polar solvents such as water can dissolve PVP.

It is soluble, for instance, in a variety of alcohols, including methanol and ethanol; moreover, in more unusual solvents, such as the deep eutectic solvent produced by urea and choline chloride [2]. It easily takes up to 40% of its mass in water from the atmosphere. When dry, transforms into a fluffy, light powder that is hygroscopic. In a solution, it readily forms films and exhibits good wetting properties [2]. Because of this, it works well as a coating or a coating additive.

Poly (Vinyl Alcohol): A man-made polymer that dissolves in water is PVA. Its idealized chemical structure is $[CH_2CH(OH)]n$. in formulations of polyvinyl acetate (PVAc) adhesives as an emulsion and thickening stabilizer, it is utilized in the sizing of textile warps in papermaking, various coatings, as well as 3D printing. It has no colour (it is white) and no smell. It is commonly offered in water solutions or beads. PVA solution may be repeatedly frozen and thawed to form very strong, ultrapure, and biocompatible hydrogels without the need for an external crosslinking agent.

Zinc Oxide Nanoparticle: Size homogeneity, surface passivation, and chemical stability are all characteristics of ZnO nanoparticles. Zinc oxide nanoparticles are the second most prevalent metal oxides after iron [3]. The price is really low, secure, and easy to make [3]. By altering the shape of zinc oxide nanoparticles produced employing different synthesis techniques, materials, or precursors, it is simple to change their physical and chemical properties. One of the inorganic groupII–IV semiconductor group compounds for applications in analytical detecting is zinc oxide nanoparticle. White powder-like in appearance zinc oxide nanoparticles, insoluble in water. Zinc oxide nanoparticles have good Stabilities of chemicals, electricity, and heat due to their 60 meV of bonding energy and a 3.37 eV energy band. (Watanabe, 2018). Additionally, the optical, electrical [3].

MATERIALS:

Polyvinylpyrrolidone (PVP) and Polyvinyl Alcohol (PVA): PVP with Mw of approximately 44.000 and PVA 98-99% hydrolysis with Mw between 31.000 and 50.000 were purchased from Central Drug House in Mumbai, India.

Zinc Nitrate ($Zn\ NO_3)_2$ with Sodium Hydroxide (NAOH): Sodium hydroxide and zinc nitrate were purchased from Hi Media, Mumbai, India.

Mint Leaves: Mint leaves are collected from the local market in Vadodara.

METHOD:

Polymeric Blend Film: A polymer blend film can be created by mixing two water-insoluble polymers or a water-insoluble polymer and a water-soluble polymer, regardless of their compatibility. Thin continuous materials, typically up to 200 m (0.008 in.) thick, are polymeric films. Over that thickness, plastic materials became known as sheets. A variety of resins can be used to create polymeric films, and each one has certain physical characteristics that make it suitable for a particular use. Melt blending, solution blending, latex blending, partial block or graft copolymers, and interpenetrating polymer networks are the five processes utilised to create polymer blends (IPN). The following manufacturing processes are used to create polymeric films, which are fundamentally manufactured derived from thermoplastic resins: using a flat die to extrude film, cooling it after, and then winding it up on a roll [4]. Cast film extrusion involves cooling or quenching the polymer melt before winding it up on a roll.

Mint Extraction: A shaker was used to combine 700 mL of ethyl alcohol with 60 g of fresh and dried peppermint for 6 hours at 70°C. Following the solvent's removal from the oil using a rotary evaporator for 30 minutes at 37°C stream, the solvent was then collected and kept in a refrigerator in a dark bottle [5].

ZnO Preparation by Co-precipitation: The substances, sodium hydroxide and zinc nitrate. Without additional processing, the compounds were utilised as they were obtained. Using 80°C heating and magnetic agitation 6 made it possible to make a 1M homogeneous aquatic precursor solution of extremely purest zinc nitrate (Zn (NO3)2.6H2O) 6 possible. The pH of the solution was discovered to be 2.9 at this point. The pH of the precursor solution was then gradually increased by the addition of 2 M aqueous NaOH solution until it reached 10. The slurry was produced as a white precipitate, and room temperature was reached by allowing it to cool naturally [6]. The precipitate was then repeatedly cleaned with distilled water and acetone, separated, and dry at 100 °C for 4 hours.

Extraction and Nano-loaded Film: The *ex-situ* approach has been used to create the PVP-PVA-ZnO solution mixture. A transparent solution was produced by stirring 5-weight percent of PVA powder with distilled water and heating it to 90 degrees Celsius. 95-weight percent of PVP powder was added to this solution. Up till a homogenous solution was generated, it was agitated. Zinc oxide

nanofiller was incorporated into the polymer blend solution at different concentrations of 2, 4, 6, and 8 wt%, distributed evenly, and dried at room temperature to produce a flexible film with a thickness ranging from 300 to 500 m. The post-loading approach was used to load the polymer film (Peppas, *et al*. 2000, Wong, & Dodou, 2017). This technique was chosen to prevent the breakdown of mint extract during making the film over the in-situ loading of the polymer. 10% stock solutions were made by mixing 1 g per extract using 10 mL of phosphate-buffered saline. Samples of (CPP) film measuring 1 x 2 cm2 were submerged in stock solutions of each mint extract for 24 hours. The samples were then individually dried at room temperature.

Chemical Test:

Physiological Fluid: In physiology, a fluids is a moist liquid that carries ions and cells required for physiological activity, as well as solutes and waste products from metabolism. Water is taken directly through meals and drinks and is the primary component of biological fluids in all creatures, including humans. To a smaller degree, it is also produced when food is oxidized during metabolism. The typical adult's daily water consumption is between 2,100 and 3,400 ml (2.2 to 3.6 quarts). Although perspiration, the epidermis, and the respiratory system all are major channels for water loss, urine is the primary way that water departs the body [7].

142mM – NaCl, 2.5mM – $CaCl_2$, NaCl 142 MW CaCl22.5 mM,

142/1000 mole.

2.5 × 147.2/1000, 0.367/ liter

142 ×58.5/1000 = 8.307/ liter

The behaviour of films swelling in the fictitious extracellular fluid with the following composition was studied. One litre of the solution included 0.367/litre of $CaCl_2$, 6.8 g of NaCl [1], 8.307/litre of sodium bicarbonate, and 3.5 g of sodium dihydrogen phosphate[1]. The pH of this solution was 7.36. A prior film sample was immersed in 100 mL of PEF at 370°C for the specified amount of time, retrieved, quickly wiped with tissue paper to remove surplus surface water, accurately weighed on an electronic balance (Denber, Germany), and then submerged once again [8] **Table 1.**

TABLE 1: SWELLING RATIO

	Swelling Ratio(g/g)		
Time(min)	**PVP/PVA(50/50)**	**PVP/PVA(60/40)**	**PVP/PVA(60/40)**
0	0	0	0
15	3.185	2.716	3.833
30	4.731	3.751	4.540
45	6.270	4.751	5.051
60	6.943	5.2	5.563
75	8	6.328	6.133
90	9.296	7.011	7.111
105	9.658	7.3	7.352
120	10.579	7.906	7.664
135	10.394	8.516	8.443
150	10.516	8.656	8.443

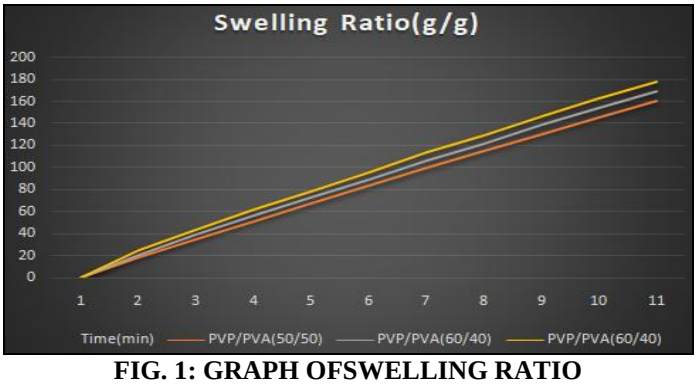


FIG. 1: GRAPH OFSWELLING RATIO

Expansion Study: As stated elsewhere, by observing the change in size of round film samples in a 4% gelatin solution, it was possible to simulate the expansion of the tissue regeneration film on the wound surface. A clear solution was created by gradually swirling 4 g of gelatin powder into 100 mL of distilled water at 85°C. A Petri plate containing 30 mL of this mixture was then filled, and it was let to freeze at 25°C overnight [5]. On the gelatin's exterior, a film sample with a known diameter was placed, and until the sample was [9] achieved a constant diameter, and the change in diameter was periodically noted. The expansion ratio is written as ER = (ER). To simulate a healing wound, the gelatin solution is employed. A film's appropriateness for usage in highly exuding wounds may be determined by observing how it expands when placed on the surface of a gelatin medium. The findings from the expansion research for the film samples PVP/PVA (50/50), PVP/PVA (60/40), and PVP/PVA (70/30) **Table 2.**

TABLE 2: EXPANSION DATA OF POLYMERIC COMPOSITES

Time (Min)	dt/d0		
	PVP/PVA (50/50)	PVP/PVA (60/40)	PVP/PVA (70/30)
0	1.0	1.0	1.0
30	1.2	1.0	1.2
60	1.5	1.1	1.4
120	1.7	1.2	1.6
150	1.8	1.2	1.7
180	1.8	1.3	1.7
210	2.0	1.3	1.7

Characterization Method:

FTIR Absorption Spectra: The innovative FTIR spectroscopy technique is based on changes in chemical compositions, chemical bonding, and modifications in functional groups during contact. FTIR research can provide details about the interaction between ions and polymers as well as molecular structure. If there is interaction, the wavenumbers and peak intensities of the function groups will change due to their vibrational modes [10]. Polymers can communicate with one another or with specific dopants through secondary hydrogen-bonding-generated bonds.

Scanning Electron Microscope Investigations: A scanning electron microscope was used to display the surface morphology of PVA and PVP-PVA blend films that were both irradiated and unirradiated (SEM). The unirradiated PVA films showed similar submicroscopic morphology, whereas small piece and phases were observed in the sem micrograph of the un-irradiated PVA/PVP and its intensity increased as the PVA concentration. The unequal distribution of PVP with various ratios and its weak interactions account for chunk and phases in PVA/PVP micrographs [11]. The enhanced cross linking following gamma irradiation, in addition hand, led to smoother regions developing in all films after gamma irradiation.

X-ray Diffraction: Cu k- radiation (= 1.540) X-ray diffraction (XRD) images were performed on the Philips diffractometer. The tube's Bragg's angle ranged from 5 to 60 degrees when operating at 30 kV. Infrared (IR) measurements were made using Nicolet's IS10 single beam Fourier transform-infrared spectrometer in the 4000 to 400 cm-1 wave number range. [Keithley 175] made DC electrical measurements with an accuracy of 2%. A programmed automated LCR metre [model Hioki 3531 Z Hitester] was used to measure the AC electrical current in the range of frequencies from 50 Hz to 3.5 MHz. Measurements for DC and AC were done among 300 and 380 K in a specially designed cell. Samples are positioned between two polished, spherical copper surfaces 12 for the best possible contact.

Thermogravimetric Analysis: Thermogravimetric analysis (TGA) that calculates weight loss about temperature is an effective analytical method for determining the thermostability of materials, especially polymers. TGA is the most important method for evaluating the thermal stability of polymers. The kinetics of the subsequent decomposition process has also been studied [4]. The kinetics of the subsequent decomposition process has also been studied. The shape of the curve is strongly influenced by the kinetics parameters order of the reaction (n) and activation energy (E).

These factors' values must be estimated to determine thermal stability. Samples are analyzed using TGA in the 30-500°C temperature range. As a function of temperature, the figure displays the usual TGA thermograms of losing weight for the present system.

For the thermogravimetric analysis (TGA), Q 50 V20 13 was employed in a platinum pan with nitrogen flowing at a 60 mL/min rate. The samples were heated at 10 °C per minute at room temperature to 600 °C. The mix's electrical conductivity and dielectric properties and composites were tested within range of 102 to 106 Hz at room temperature using a fully automated Hewlett-Packard LCR meter [13].

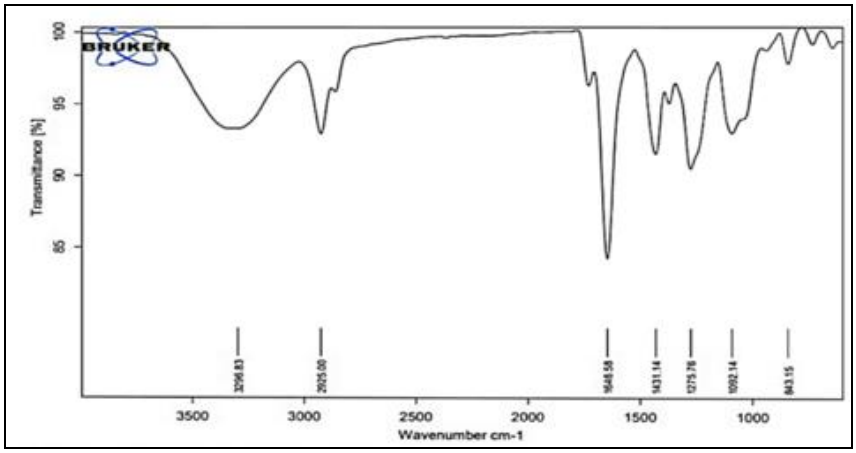
UV-Vis Absorption Spectrum: We looked at the UV-vis absorption spectra of polymeric in water using a Jasco V 550 spectrophotometer. The nanoparticles' dimensions significantly impact how materials' whole characteristics are altered. To learn more about the materials' properties, semiconducting nanoparticles' size development becomes extremely important. Examining the optical characteristics of nanosized particles is frequently done using the UV-visible absorption spectroscopy method. The figure displays the ZnO nano powder's absorption spectrum. At roughly 355 nm, it shows a significant absorption band. A peak of excitonic absorption is seen at about 258 nm because of the presence of ZnO nanoparticles, which are placed significantly underneath the wavelength of the band gap of 366 nm (Eg = 3.46 eV). The intense acute absorption of ZnO makes it abundantly evident that the nanoparticle dispersion is monodisperse.

RESULT AND DISCUSSION:

FTIR Absorption Spectra: The melting of PVA and PVP led to forming of a distinct C=N (pyridine ring) band equivalent to PVP at 1548 cm $^{-1}$ [7]. The OH band was somewhat shifted due to the hydrogen link between the PVP carbonyl and PVA hydroxyl groups. Similarly, when the PVA component in the PVA/PVP films grew, The C-N stretching band was found to have decreased somewhat from 1287 cm-1 to 1236 cm^{-1} [7]. The conjugate between the N and C=O groups explains this change [7]. The existence of the C=O stretching band in FTIR spectroscopy proves that PVA/PVP mixes have a semi-crystalline structure. As can be observed, gamma radiation widened and intensified the hydroxyl group peak [14].

At room temperature, the pure PVA/PVP blend's FTIR spectra are shown in the 400–4000 cm-1 region. The PVA and PVP spectra contain practically all of the significant bands. The spectra match those that have already been reported nicely. On the captured absorption spectra, distinct bands representing the bending and stretching vibration of the functional groups inside the samples can be detected. The positions of the absorptions and their assignment for the characteristic bands are listed in the following table. The spectra show changes in certain bands and differences in the intensity of other bands when pristine blend films are considered. This demonstrates the blend's significant interaction. The absorption peak at 1647 cm^{-1} was ascribed to the stretching vibration of the carbonyl group (C=C), whereas the peak at 1088 cm^{-1} was assigned to the carbonyl group (C=O) [15]

Fig. 2.



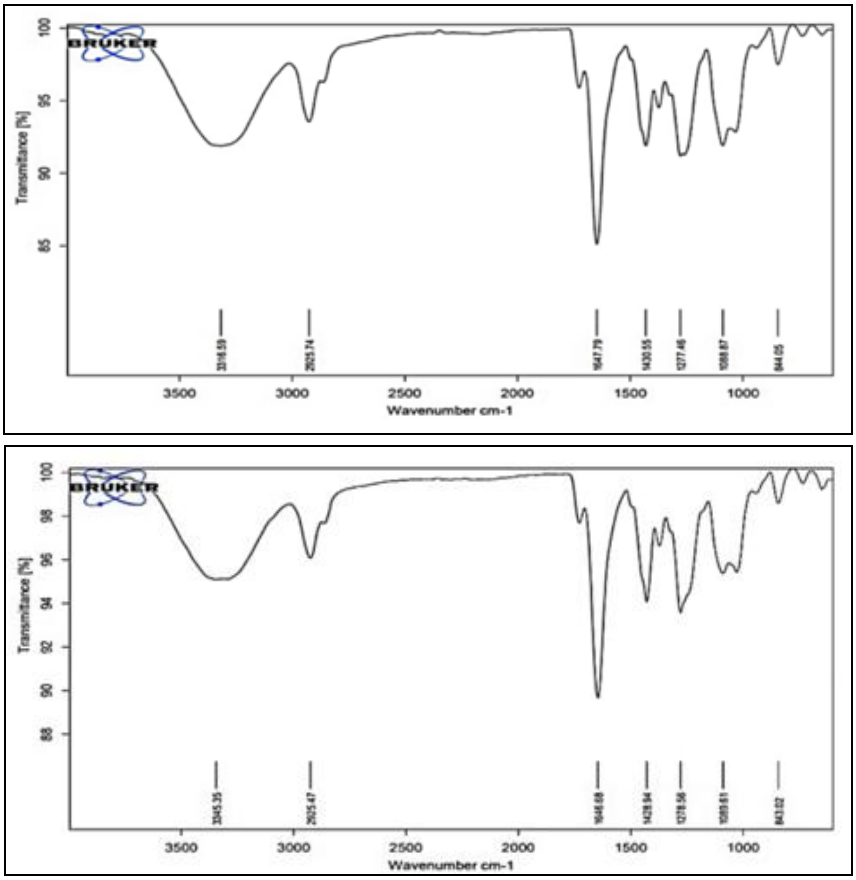


FIG. 2: FTIR SPECTRUM OF PVP/PVA COMPOSITE FILM WITH DIFFERENT WEIGHT RATIOS

Assignments of the PVA/PVP blend's IR characteristic peaks:

Wave number (cm^{-1})	Assignment
844.05	OH stretching
1088.87	C = O stretching
1277.46	C – O – C stretching
1430.55	C = C stretching
1647.79	C = C stretching
2925.74	CH2 asymmetric stretching
3316.59	OH stretching

X-ray Diffraction: The figure displays the X-ray diffraction (XRD) pattern of the prepared PVP/PVA polymeric film. This XRD pattern was analyzed using the PAN analytical High Score Plus v 3.0e program Software. The crystallite size, phase composition, and crystalline structure of ZnO nanopowder are identified.

The X-ray diffraction pattern of ZnO nanopowder is shown in figure. The figure depicts the 50/50 composite films. The patterns display peaks typical of 50/50 PVA/PVP mix films, with centers around 2 = 12.8 and 20.1 **Fig. 3.**

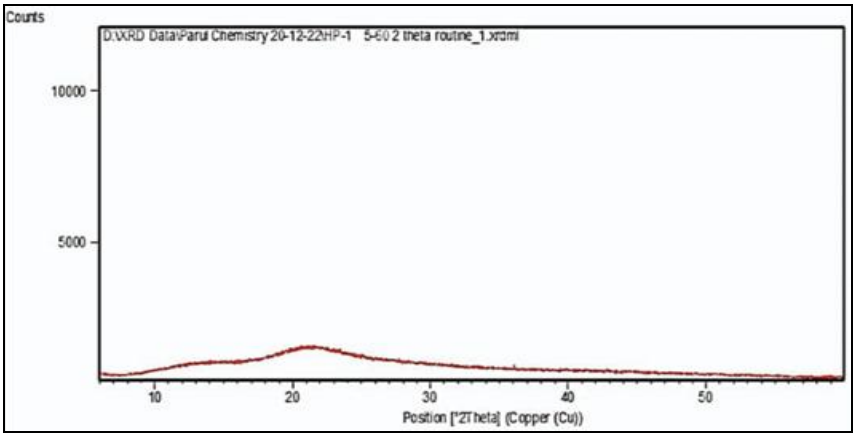


FIG. 3: X-RAY GRAPH OF THE COMPOSITE FILM

Scanning Electron Microscope Investigations: The microstructure and dispersion of the Nano filler, which improves the shape of the film, are assessed by SEM. SEM photos of the PVP-PVA polymer film in its natural mix and the PVP-PVA-ZnO nanocomposites are both obtained at a magnification of 10kX. It is evident that there is a high degree of orientation and that the bigger, more elliptical, and floral-shaped ZnO grains are spread rather evenly [10].

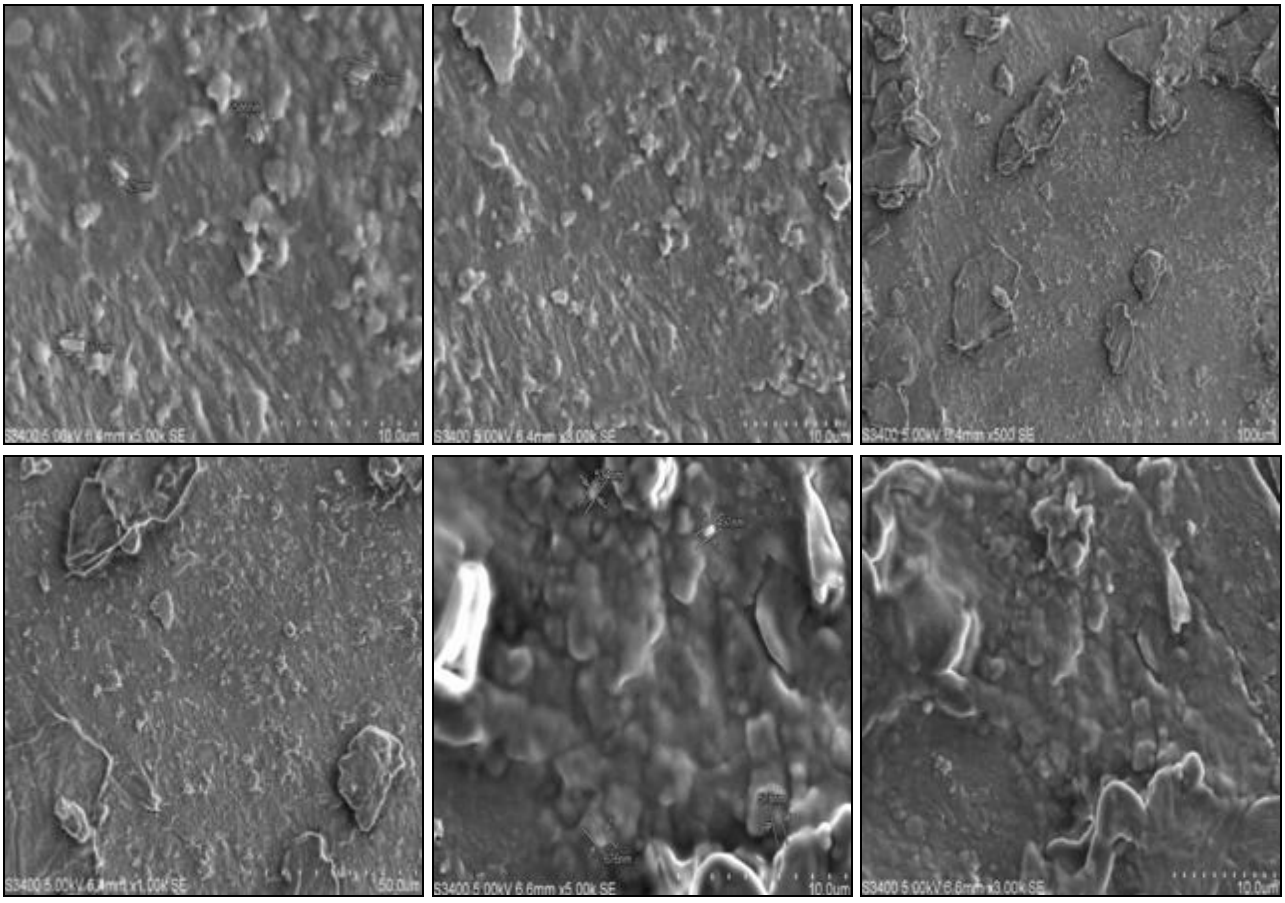


FIG. 4: SEM PICTURES OF THE OUTER LAYER OF COMPOSITE FILM

UV-Vis Absorption Spectrum: Spectrum of UV-Vis Absorption. A material's overall qualities can be changed by changing the size of the particles. Therefore, semiconducting nanoparticle size development becomes crucial to understanding the properties of the materials. Examining the optical characteristics of nanosized particles is frequently done using the UV-visible absorption spectroscopy method. At roughly 370 nm, it shows a significant absorption band. An excitonic absorption peak is seen at about 258 nm due to the presence of ZnO nanoparticles, which are situated far below the band gap wavelength of 358 nm (Eg = 3.46 eV). The intense acute absorption of ZnO makes it abundantly evident that the nanoparticle dispersion is monodisperse. UV-vis absorption spectra of ZnO nanoparticles show an absorption peak at around 370 nm. The semi-crystalline nature of the materials and the n p* transition can be used to explain the spectra's absorbance at approximately 235 nm **Fig. 5.**

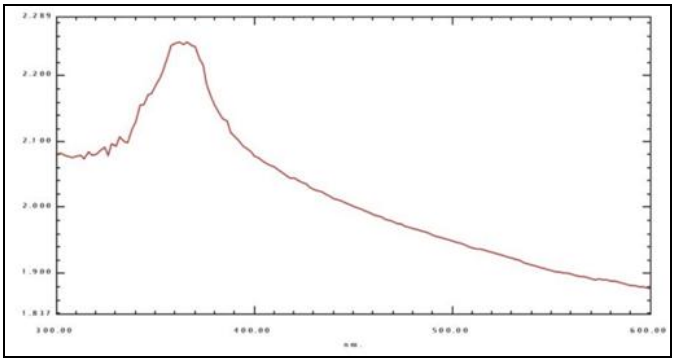


FIG. 5: THE ULTRAVIOLET-VISIBLE SPECTRUM

Thermogravimetric Analysis: When exposed to TGA thermograms in an air atmosphere, PVA and PVP show almost the same overall losing weight in a PVA/PVP mix film, showing that both polymers have the same thermal stability **Fig. 6.**

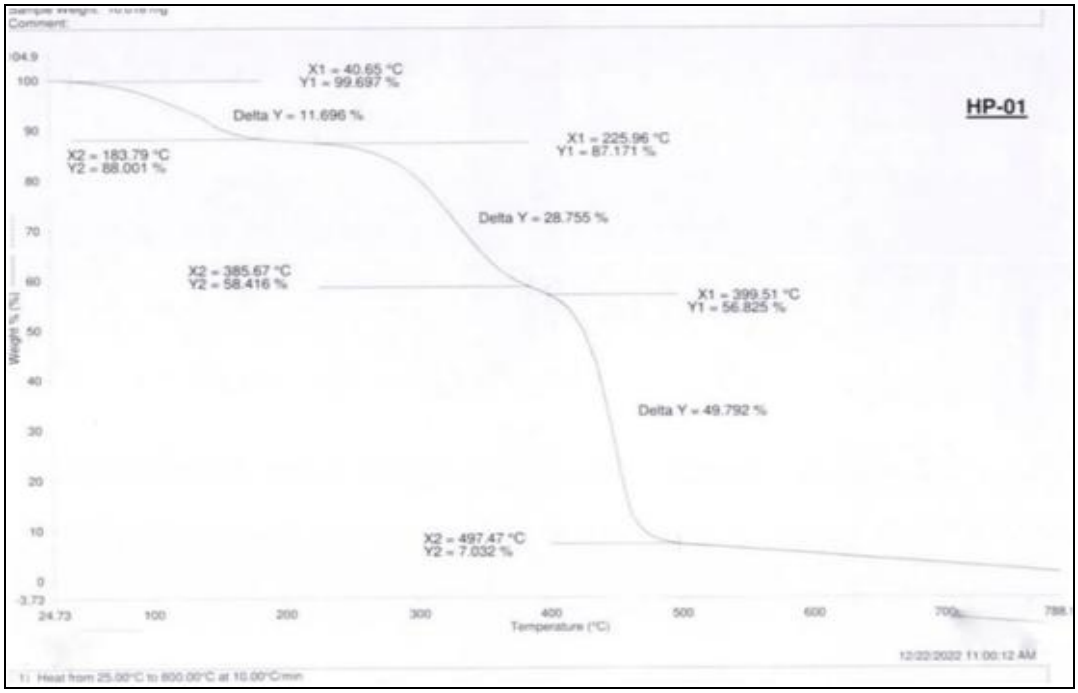


FIG. 6: TGA GRAPH

We may infer that blending narrows the temperature range in which this phase takes place for both PVA and PVP. This might be because the mixture of PVA and PVP formed linked hydrogen bonds, which would allow the solvent to evaporate more readily at lower temperatures. Films made of PVA/PVP mix begin and conclude their first breakdown process at temperatures lower than those of PVA and PVP (Except film AB5, which begins at a greater temperature than all the others).

The drop in temperatures at which all PVA/PVP films start and finish their first and second breakdown processes does not follow any systematic pattern when compared to PVA and PVP. As the proportion of PVP in PVA/PVP blend films rises, more water will be absorbed as PVP is more hydrophilic than PVA and decreases PVA's crystallinity.

Additionally, we find that, when comparing to PVA, the weight loss at the first decomposition phase of all PVA/PVP blend films increases with the ratio. This is because the losing weight at the first decomposition step of PVA/PVP blend films indicates the elimination of solvent and oligomers.

Antibacterial: The bactericidal efficacies of two ZnO/PVA-PVP and Mint Extract blends, as well as pure PVA-PVP blend, were compared using a modified Kirby Bauer method Gram-positive *S. aureus* and Gram-negative *E. coli* were both susceptible to the antibacterial effects of the ZnO/PVA-PVP and Mint Extract combo after 24 hours at 378°C. The width of the inhibition zone is somewhat bigger for the mix of 1.0 wt% ZnO/PVA-PVP and mint extract than it is for the 0.2 wt% ZnO/PVA-PVP and mint extract sample see Fig. The pure PVA-PVP mixture has no capacity to inhibit when used as a control.

Elemental zinc has been thought to behave as a contact-active substance or as a release method for zinc oxide to have antibacterial properties. The ZnO/PVA-PVP and Mint Extract mixture appears to be solely contact-active in the current investigation. The Kirby Bauer technique's limitations as a quantitative method to assess antibacterial activity include the possibility that the creation of secondary ZnO compounds reduced the zinc oxide's capacity to diffuse on an agar plate.

In order to quantitatively assess the antibacterial activity of the ZnO/PVA-PVP and Mint Extract combination, the LB medium technique was introduced.

The samples studied included pure PVA-PVP mix, Zno/PVA-PVP, and mint extract. As can be seen in figure, increasing the ZnO level in the blend improved the blend's antibiotic effectiveness against *E. coli* as measured by the inhibition ratio

[12]. When the ZnO concentration reached 1.0 wt%, the inhibition ratio increased to 90%. Mild antibacterial characteristics shared by menthol and mint extract make them both efficient against both Gram-positive and Gram-negative bacteria. Additionally, antiviral and fungicidal properties of mint have been discovered.

Mint leaves' potent anti-inflammatory effects may effectively cure dry, itchy skin as well as cuts, wounds, and insect bites. Juice from a mint leaf may be extracted and applied to dry skin or wounds to help soothe irritated and burning skin **Fig. 7.**

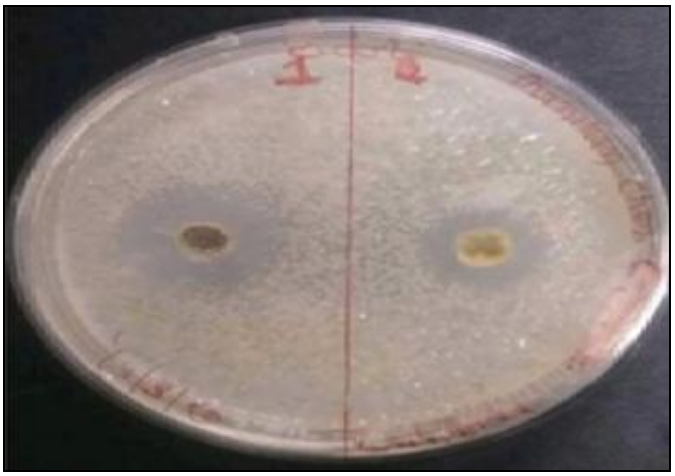


FIG. 7: ANTIBACTERIAL ACTIVITY

CONCLUSION: Co-precipitation has been used to manufacture 32nm pure phase ZnO successfully. Adding ZnO to the polymer causes the patterns to crystallise more. ZnO's interaction with the polymer is confirmed by the absorption peak of ZnO in the composite films. The conclusion is that adding nano ZnO filler improves the smoothness of the surface. The homogeneous ZnO nanofiller dispersion in nanocomposite films is seen. SEM pictures also display small aggregates of the particles. The absorption edge shifts toward a shorter wavelength (blue shift) with a drop in ZnO Nanoparticle concentration. An affordable and effective solid-state solar cell or LEDs may be made using the polymer blend and zinc oxide nanofiller. A mix of natural medicine Phudina extract and PVP/PVA with ZnO nanoparticle can be used to treat wounds.

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CONFLICTS OF INTEREST: Declared None

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