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## CHEMICAL ANALYSIS AND MORPHOLOGICAL STUDIES OF POLYMERIC COMPOSITE FILM

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### ABSTRACT

By esterifying polyvinyl alcohol and gelatin, a blend hydro gel was attempted to be prepared in the current experiment. The traditional solution-casting technique was used to further transform the blended hydro gel into films. FTIR, X-ray diffraction, SEM, TGA analyses were used to analyses these films. Pf swelling test, MVTR and WVTR behavior of films were also studied. The free carboxylic group of gelatins had been completely esterified, according to the FTIR spectra of the polymer mix film. The TGA results that indicate melting point of polymeric films at different higher temperatures this way other analysis like SEM and XRD also shows the properties of the film. Gelatin and PVA interact to alter the crystallite's properties and degree of crystallinity. Gelatin was primarily responsible for the blend film's crystallinity. The segment density within the molecular coil increased, according to the viscosity comparison. The findings demonstrated the modifications in the gel's characteristics and improved gel formation during the material's viscoelastic phase. The mix film was strong enough and capable of holding water.

**Keywords:** PVA, Gelatin, FTIR, TGA, XRD, SEM, PF swelling

### INTRODUCTION

Biomaterials communicate with living things crucial to manage a biomaterial's surface by means of their surfaces. Therefore, it is characteristics to make the substance

"biocompatible," or to ensure good integration with the host tissues. In addition to offering great flexibility Organic thin films and coatings, particularly those made of polymers, are highly desired as biomaterial coatings because they have mechanical properties that are similar to soft biological tissues. This makes them ideal for controlling tissue-biomaterial interactions in the chemical groups that can be incorporated at the surface [6]. The general interest in organic thin films might also be attributed to their relatively straightforward processing. Polymer coating of biomaterial surfaces is possible utilizing.

#### **PVA (polyvinyl alcohol)**

Because it is water-soluble and biodegradable, poly-vinyl alcohol (PVA) is a desirable material for bio plastics. PVA is a popular bio plastic that has good qualities, such as strong oil and chemical resistance, and is stable in virtually all organic solvents. PVA is also appropriate for use as a paper adhesive and in packaging. A polymer made from polyvinyl acetates by substituting hydroxyl groups for the acetate groups. It is utilized in the production of cosmetics, artificial sponges, surface coatings, pharmaceutical aids, ophthalmic lubricants, and other items.

#### **Gelatin**

When cooled below 35 °C, it forms gel and is largely utilised as a gelling agent in the food,

drug, and cosmetic industries [4]. Transparent, elastic, and thermo-reversible are all characteristics of gelatin gels. Many researchers have employed this strategy in the creation of controlled release medication delivery devices. Films made of gelatin are transparent, elastic, robust, and oxygen permeable. The drying temperature has an impact on the characteristics of gelatin films. Due to its superior film-forming qualities and quick disintegration in stomach juices, gelatin is still the preferred material for making capsules [4]. Gelatin has excellent gas barrier qualities and is extremely hydrophilic.

#### **Materials**

Polyvinyl alcohol (PVA) and Gelatin is purchased from HI Media Laboratories Pvt. Ltd- Mumbai (INDIA) Concentrated Hcl is purchased from Loba Cheme Pvt. Ltd.- Mumbai (INDIA).

#### **Method**

Preparation of PVA and Gelatin film : First take 25ml DL water in a beaker , then put it on a magnetic stirrer and heat it to 60 degree c and 300-350 rpm , then take precise measurements of gelatin and PVA in it and add it to the water and its measurements are respectively 90:10 , 85:15, 80:20, 60:40 Add in 4 different beaker and finally after equal mixing of them add 1 ml HCl (conc) and let it settle for 30-35 min and finally take it out in

Petridis and dry it in oven and then separate it from the Petridis.

### Chemical analyses

#### Pf swelling test

The pf swelling test of film was studied by prepared 1 liter solution of NaCl – 8.307 g/liter and 1 liter solution of CaCl<sub>2</sub> – 0.367 g/liter. Mixed two solutions of NaCl and CaCl<sub>2</sub> and then pre-weighed 4 different film was placed into 100 ml of pf solution at 37°C and it was removed from the water at regular intervals, weighed precisely with an electronic scale (produced by Denber, Germany), lightly rubbed with tissue paper to remove any extra surface water, and then placed back into the water. The following equation is used to calculate the pf swelling ratio:

$$SR = \frac{(M_t - M_0)}{M_0} \quad \text{g/g}$$

Where, M<sub>0</sub> and M<sub>t</sub> respectively, stand for the initial mass and the mass at different points in time.

#### MVTR (Moisture vapor transmission rate)

MVTR was performed by following method. To examine the kinetics of moisture absorption, a round metallic block was positioned in the middle of a plastic container with the top of the block above the level of the saturated KNO<sub>3</sub> solution [1]. Now the steel block was topped with an aluminium crucible. The crucible was covered with a piece of fully

dry 4 different films that had been pre-weighed; the jar's lid was then snugly fastened; the jar was then put in an incubator with the temperature set at 37°C. At certain intervals, the film was taken out, accurately weighed using a digital balance, and then returned to the jar [1]. the mass measurements persisted until equilibrium was reached and were represented as g/g dry films [1]. Each value was entered into the equation that follows.

$$\frac{1}{(m - m_0)} = \frac{1}{k(m_e - m_0)} + \frac{1}{(m_e - m_0)}$$

Where m<sub>0</sub> and m<sub>e</sub> represent the masses of the dry and completely equilibrated films, while m stands for the mass of the hydrated film at different times [1].

#### WVTR (Water vapor transmission rate)

WVTR was performed by following method. After covering the test film sample's mouth with cello tape and adding 10 ml of distilled water, the reusable cup's mouth was given a diameter of 30 mm [1]. The film has an effective area of around 3.9 10<sup>-4</sup> m<sup>2</sup>. At certain times, the cup was removed, properly weighed with a digital weighing balance [1].

$$MVTR = \frac{24 M}{a \cdot \Delta t} \quad [1]$$

Where, M= loss of water (g)

Δt = time duration

a = Effective transfer area per m<sup>2</sup>. [1]

#### Characterization of film

**1 FTIR:** To capture the Fourier Transform Infrared (FTIR) spectra, an FTIR spectrophotometer was employed [10].

**2 XRD (X-ray Diffraction):** Analyzing X-ray diffraction is a method for figuring out a substance's crystallographic structure. When using XRD, a material is exposed to incoming x-rays, and the intensity and scattering angles of the x-rays that escape are measured [5].

**3 TGA (Thermogravimetric analysis):** TGA is a technique for material analysis that involves heating a sample under strict supervision and determining how much mass changes with temperature or time. As the material's constituent parts gradually volatilize over time, temperature and weight loss are noted. Because TGA testing can measure weight loss at very high temperatures, it is a useful option for evaluating polymers. However, some polymers may stand temperatures of 300°C in air and 500°C in inert gases without deteriorating. Polymers typically melt at

roughly 200°C before breaking down. These polymers can be examined by TGA as well [6].

**4 SEM (scanning Electron Microscopy):** A concentrated source of electrons is produced by an electron microscope known as a scanning electron microscope (SEM) to scan a surface and create images of the material. The surface structure and chemical composition of the sample are shown by the different signals produced when electrons interact with the sample's atoms.

### Result of chemical analysis

#### Pf swelling behavior of films

It is crucial to look into the ability of wound dressing films to swell [1]. By absorbing a lot of moisture, the film tries to keep the wound bed dry from the exudates while simultaneously releasing the contained substance to stop bacterial infection. **Table 1**, shows the pf swelling of films sample PVA/Gt (90/10), PVA/Gt (85 /15), PVA/Gt (80/20) and PVA/Gt (60/40) is shown in **Figure 1**.

**Table 1: pf swelling of films sample PVA/Gt (90/10), PVA/Gt (85 /15), PVA/Gt (80/20)**

Time(min)	Swelling Ratio(g/g)			
	PVA/ Gt (90/10)	PVA/Gt (85/15)	PVA/Gt (80/20)	PVA/Gt (60/40)
0	0	0	0	0
30	0.267	0.364	0.309	0.219
60	0.299	0.381	0.312	0.258
90	0.328	0.394	0.316	0.260
120	0.334	0.402	0.321	0.284
150	0.338	0.408	0.329	0.294
180	0.343	0.414	0.335	0.296
24hr	0.360	0.430	0.343	0.350

The result shows that sample PVA/Gt (85/15) had a larger water uptake than the other samples and sample PVA/Gt(90/10) had a smaller water uptake than the other sample. According to a power fractional law it derived that water uptake is dependent on time.

$$\frac{M_t}{M_\infty} = k t^{n \dots}$$

Where,  $M_t$  and  $M_\infty$  are the masses of the expanded film at time  $t$  and in the actual dry state, where  $k$  and  $n$  are swelling exponent and film characterization constant. The equation is written in logarithmic form as

$$\ln F (M_t/M_\infty) = \ln k + n \ln t$$

Table 2: Data displaying  $\ln F$  against  $\ln t$  values at various time for different samples PVA/Gt (90/10), PVA/ Gt (85 /15), PVA/ Gt (80/20) and PVA/ Gt (60/40)

ln t	ln $M_t/M_\infty$			
	PVA/Gt (90/10)	PVA/Gt (85/15)	PVA/Gt (80/20)	PVA/Gt (60/40)
3.4	-0.29	-0.17	-0.1	-0.47
4.09	-0.18	-0.12	-0.095	-0.31
4.49	-0.093	-0.088	-0.082	-0.29
4.78	-0.075	-0.067	-0.066	-0.2
5.01	-0.062	-0.057	-0.041	-0.18
5.19	-0.048	-0.038	-0.023	-0.17

The first order kinetic model is employed to describe the rate of water absorption as follows [2]:

$$dM_t/dt = k_1 (M_\infty - M_t)$$

Where,  $k_1$  represents the first order rate factor for expansion [2].

Limits=0,  $M_t = 0$ ; and  $t=t$ ,  $M_t = M_t$ , we get,

$$\ln (1 - M_t/M_\infty) = -k_1.t [2]$$

This means that  $\ln (1 - M_t/M)$  against  $t$  graphs should be linear with a slope of  $k_1$  [2]

Table 3: Data on first order kinetics for different samples PVA/Gt (90/10), PVA/Gt (85 /15), PVA/Gt (80/20) and PVA/Gt (60/40)

Time(min)	ln $1 - M_t/M_\infty$			
	PVA/Gt (90/10)	PVA/Gt (85/15)	PVA/Gt (80/10)	PVA/Gt (60/40)
0	0	0	0	0
30	-1.356	-1.871	-2.331	-0.983
60	-1.775	-2.489	-2.403	-1.336
90	-2.42	-2.12	-2.541	-1.358
120	-2.628	-2.732	-2.747	-1.669
150	-2.795	-2.973	-3.198	-1.797
180	-3.053	-3.291	-3.758	-1.869

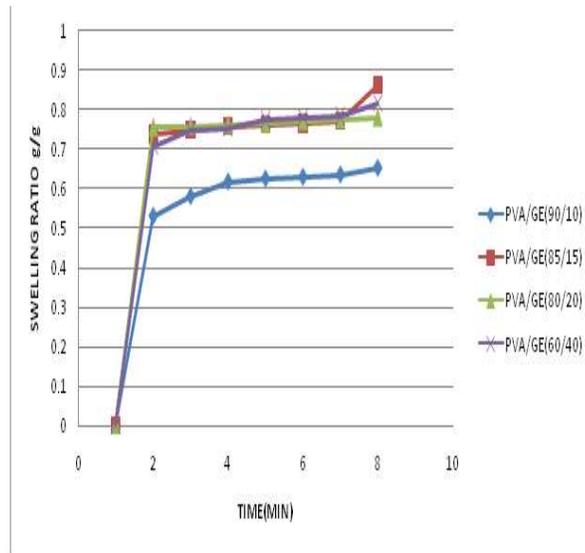


Fig.1 graph of swelling ratio against time and PVA/GELATIN (90/10), PVA/GELATIN (85/15), PVA/GELATIN (80/20), PVA/GELATIN (60/40).

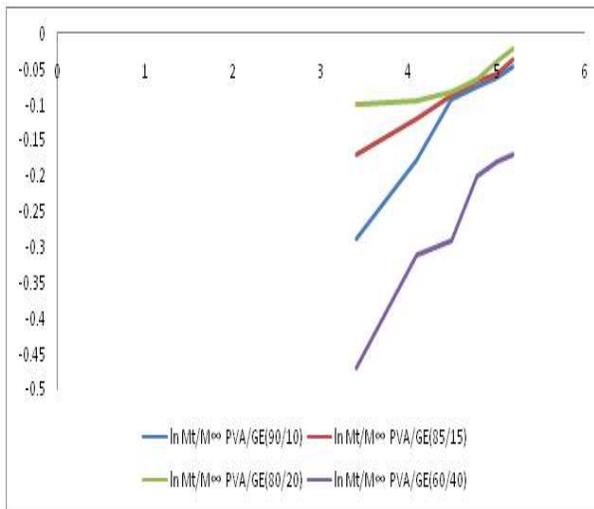


Fig. 2 Graph of ln t against ln M/M∞ for film sample PVA/GELATIN (90/10) , PVA/GELATIN (85/15), PVA/GELATIN (80/20), PVA/GELATIN (60/40).

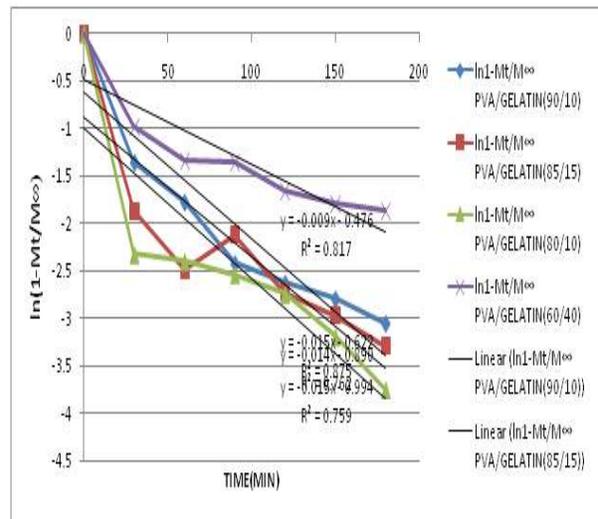


Fig.3 showing graph is on first order kinetics for different samples PVA/GELATIN (90/10), PVA/GELATIN (85/15), PVA/GELATIN (80/20), PVA/GELATIN (60/40).

Table 4: MVTR (Moisture vapor transmission rate) value for different sample films PVA/G t(90/10), PVA/ Gt (85 /15), PVA/ Gt (80/20) and PVA/ Gt (60/40)

Time (min)	Moisture uptake (g/g) of samples			
	PVA/Gt (90/10)	PVA/Gt (85/15)	PVA/Gt (80/20)	PVA/Gt (60/40)
0	0	0	0	0
15	0.022	0.016	0.022	0.028
30	0.025	0.022	0.029	0.03
45	0.029	0.026	0.035	0.035
60	0.034	0.031	0.041	0.037
75	0.039	0.036	0.049	0.039

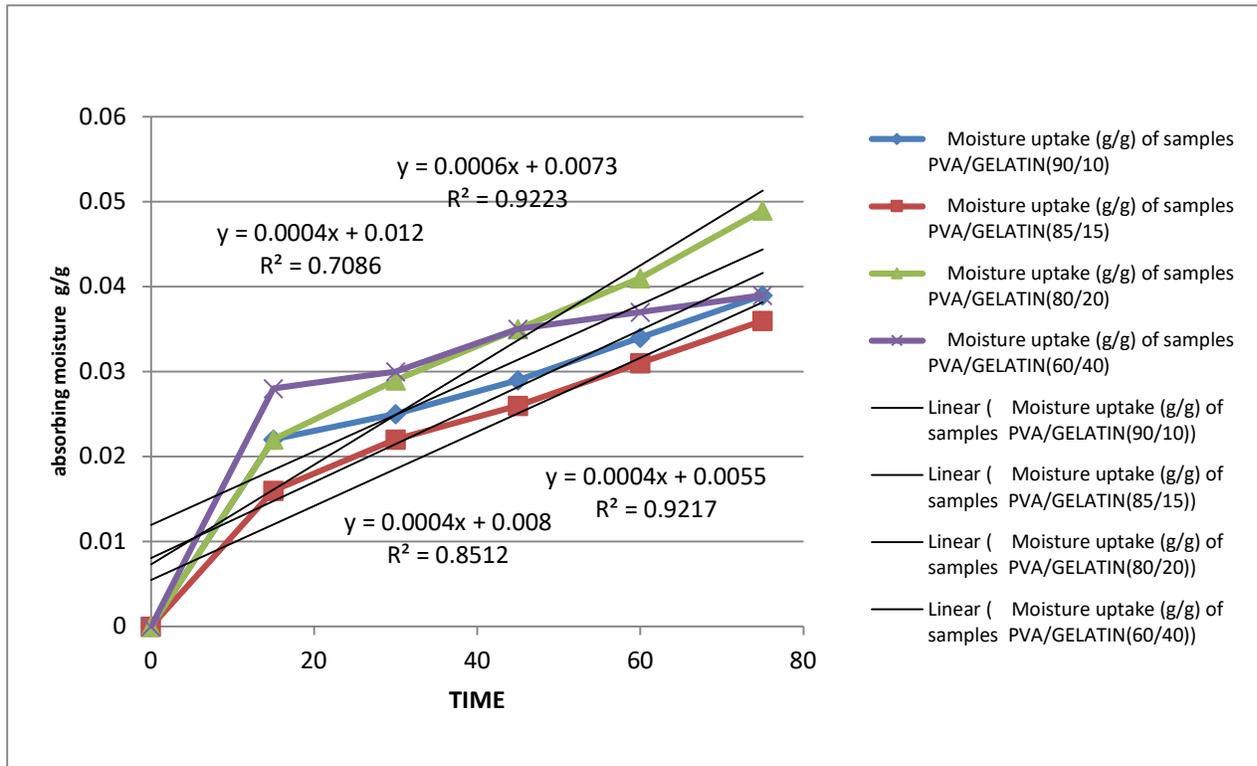


Figure 4 show MVTR for different film PVA/Gt (90/10), PVA/Gt (85/15), PVA/Gt (80/20), PVA/Gt (60/40). All sample PVA/Gt (90/10), PVA/Gt (85/15), PVA/Gt (80/20) and PVA/Gt (60/40) show reduce moisture uptake.

Table 5: Data of various film sample PVA/Gt (90/10), PVA/ Gt (85 /15), PVA/ Gt (80/20) and PVA/ Gt (60/40) to study WVTR

Time(h)	Moisture loss (g)			
	PVA/Gt90/10)	PVA/Gt(85/15)	PVA/Gt(80/20)	PVA/Gt(60/40)
0	0	0	0	0
24	0.00094	0.00098	0.00096	0.00096
48	0.00048	0.00049	0.00047	0.00047
72	0.00031	0.00033	0.00031	0.00031
96	0.00023	0.00024	0.00023	0.00023
120	0.00018	0.00019	0.00018	0.00018
144	0.00015	0.00016	0.00015	0.00015
168	0.00013	0.00013	0.00013	0.00012
192	0.00011	0.00012	0.00011	0.00011
216	0.0001	0.0001	0.0001	0.0001
720	2.396	2.473	2.353	2.327

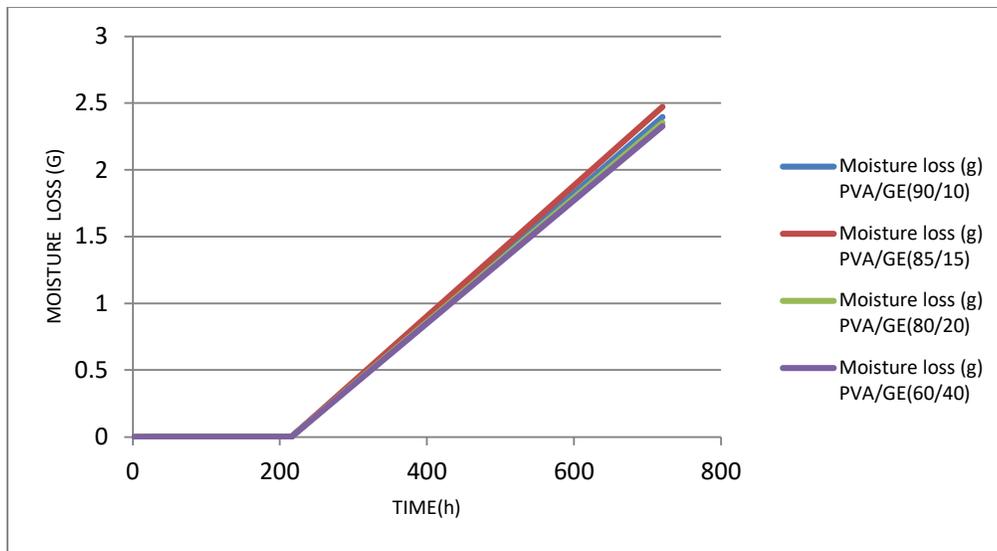


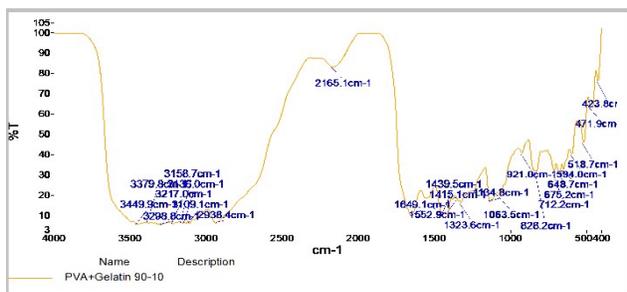
Figure 5: WVTR of different sample films PVA/Gt(90/10), PVA/ Gt(85 /15), PVA/ Gt(80/20) and PVA/ Gt (60/40)

Result of characterization of films

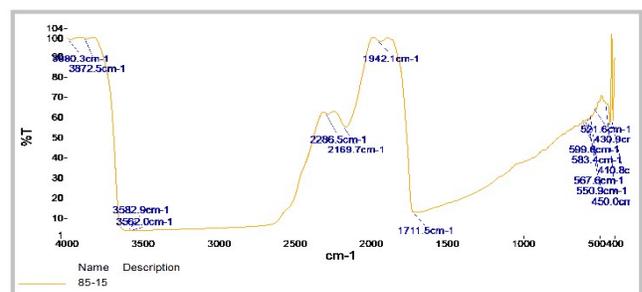
Result of different films in FTIR

FTIR analysis is as maintained in Figure 6 the functional group in a combination can be analysed with the help of FTIR analysis. The

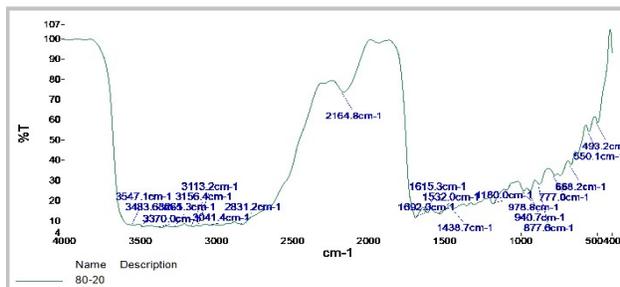
films exposed to gamma radiation displayed a positive peak, and this standout peak demonstrates how the crosslinking process enhances the chemical interaction between gelatin and PVA [7].



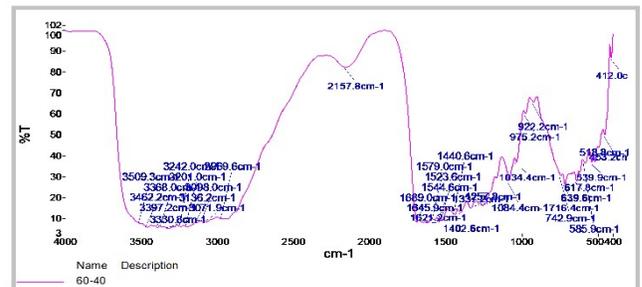
(A)



(B)



(C)



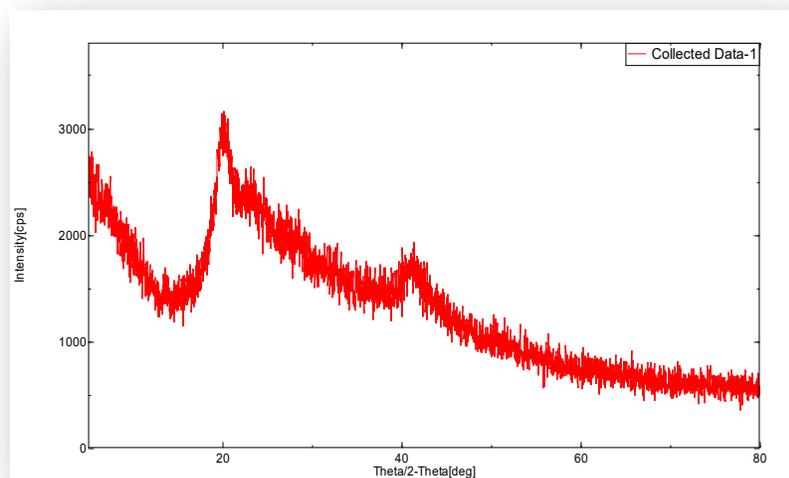
(D)

Figure 6: Showing FTIR spectrum of films sample (A) PVA//Gt (90/10) (A), PVA/ Gt (85 /15) (B), PVA/ Gt (80/20) (C) and PVA/ Gt (60/40) (D)

### Result of the PVA/Gt film in XRD.

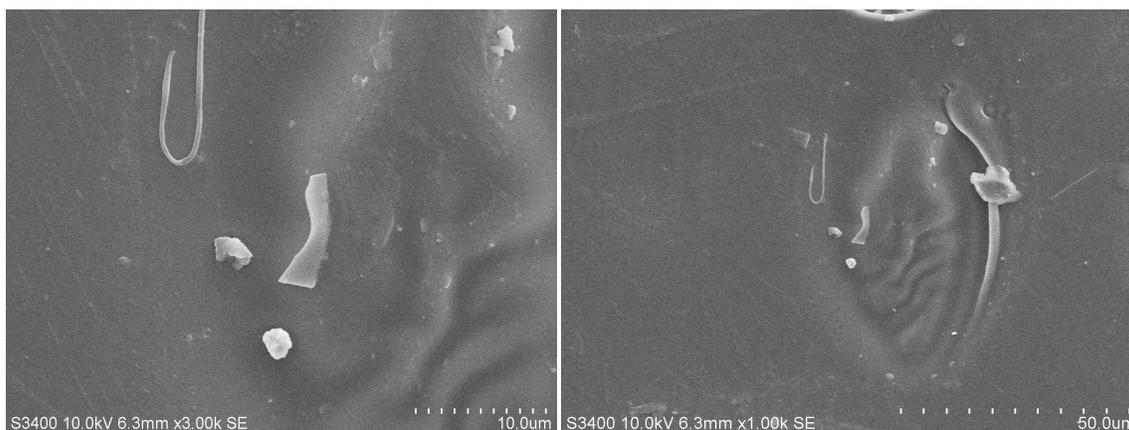
XRD analysis is as maintained in **Figure 7** as can be seen in the image above, X-ray diffraction reflections were used to examine the phase of gelatin. In conclusion, gelatin

produces well-defined signals with smaller peak widths while having an amorphous phase. The information gathered agrees with the conclusions reached by other authors [8].



**Figure 7: Showing XRD spectrum of films sample PVA/Gt(85/15)**

7.3. Result of the PVA/Gt film in SEM analysis is as maintained in **Figure 8**.



**Figure 8: Showing SEM spectrum of film sample PVA/Gt (85/15)**

From this analysis we observe the surface and the other properties of this film. SEM photos of the nanocomposite's surface and interface (fractured surface). Because of the homogenous distribution of graphene fillers

within the PVA-Gt matrix, the clear surface of the composite implies that graphene fillers are missing on the surface of the composite [9].

### Result of the PVA/Gt film in TGA

TGA analysis is as maintained in **Figure 9**. As a consequence, the intermolecular cross-linking activity produced highly compatible

impactful mixed polymers, which contributed to the increased thermal stability reported for TGA and its descendant blended samples.

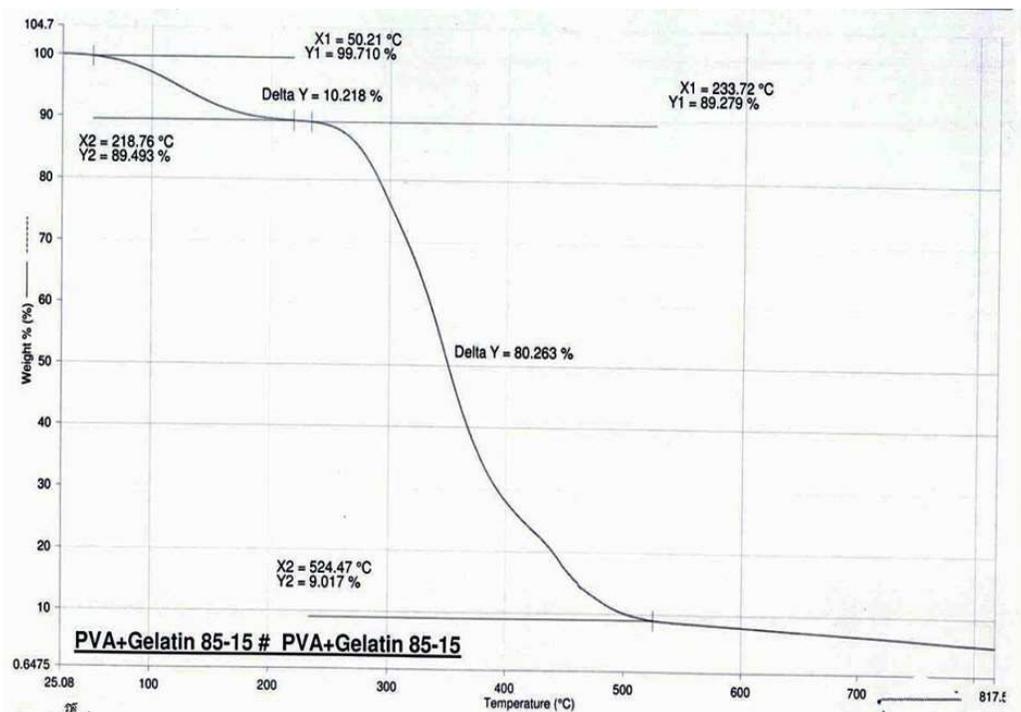


Figure 9: Showing TGA spectrum of film sample PVA/Gt (85/15)

## CONCLUSION

The hydro gel films made from a PVA/Gt blend were prepared by esterifying the carboxylic group of the gelatin with the hydroxyl group of PVA. The gelatin's entire carboxylic group has been esterified, according to the FTIR study, but the free amino groups are still present. The XRD data show that structural repositioning occurs as a result of gelatin's interaction with PVA chains, which causes crystallinity to decrease as gelatin content rises. The PVA/Gt blend

solution's refractive index decreases as the gelatin component rises. Blends of PVA and gelatin became less viscous as the amount of gelatin in them increased. The films can be employed as wound covering since they can withstand some frictional pressures from daily activities, according to their tensile strength. Therefore, this cross-linked gel could be utilized in synovial joints as artificial cartilage. The gel produced was discovered to be extremely absorbent, making it suitable for use in a number of medical procedures,

including the delivery of drugs and moist wound dressings.

**Acknowledgment:** I wish to thank PNP Analytical Solution for providing the facilities of FTIR, TGA, SEM, XRD and many other analysis.

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