

## Study of thermogravimetric analysis and some thermal properties of a binary mixture of polyester and pineapple peel powder

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### Abstract:

In this study, agricultural waste was minimized by incorporating small amounts of pineapple peel powder into unsaturated polyester resin, allowing an investigation into how this addition influences the resin's thermal properties. The thermal characteristics were assessed before and after reinforcement by adjusting the pineapple peel powder weight ratios (5%, 10%, 15%). Techniques such as Thermogravimetric Analysis (TGA) and Infrared Gas Analysis (IGA) were utilized to examine the resin's properties at various temperatures (25, 55, 85, 125, and 155 °C).

The weight data at 350 °C, denoted as {Wt%}{350}, were found to align closely with the TGA results, suggesting that the unsaturated polyether ester content in the pineapple peel powder contributes to slight heat sensitivity. These findings support TGA data as a reliable indicator of thermal stability.

Furthermore, the initial decomposition temperature (IDT) did not exceed 420 °C across treatments, while the complete decomposition temperature (CDT) ranged from 330 to 720 °C, with an average residual weight at 350 °C of less than 55%. Notably, the IDT and CDT results for polymer composites containing 15% pineapple peel powder showed higher stability compared to lower concentrations.

**Keywords:** gravimetric thermal, polyester, polymeric composites, pineapple peel powder.

### Introduction:

Due to scientific advancements, there has been a rising demand for polymeric materials with specific properties that cannot be achieved from a single source. Consequently, extensive efforts have been made to combine different materials to produce polymeric composites with the required industrial specifications (1). Composite materials, also known as superimposed materials, are heterogeneous systems comprised of two or more immiscible substances. Their purpose is to achieve enhanced properties that conventional materials cannot provide.

These materials offer numerous advantages, including light weight, thermal and electrical insulation, mechanical and chemical resistance, and design flexibility (2). Over the past century, the scientific and industrial significance of polymers has increased substantially. Global statistics estimate that over 40% of engineers and 20% of scientists are engaged in the plastics industry and its associated technologies (3). Many modern industries require materials with combined attributes, such as lightness and durability, which are not naturally found in traditional materials like metal alloys and ceramics (4, 5). Additional benefits of polymeric composites include ease of manufacture, resistance to oxidation, and compatibility with various solutions, such as acids and bases, as well as user-friendly properties.(7 ,6)

These composite materials have extensive applications, with an annual utilization exceeding two million tons in structural products such as tanks, pipes, vehicle components, boat hulls, and aircraft panels, all of which benefit from enhanced strength and rigidity (8). Unsaturated polyesters, in particular, are frequently reinforced with fillers, which may be either organic or inorganic (9).

In recent years, there has been an increasing focus on organic fillers due to their availability and sustainability compared to raw materials. Commonly used organic fillers include animal bones, shells, eggshells, walnut shells, and various plant husks (10). Inorganic fillers, such as iron filings (11) and glass powder (12), are also widely utilized to reinforce polymeric structures.

### **Materials and methods:**

Initialization: The initialization of raw materials involved:

Unsaturated Polyester Resin

Preparation of Additive (Pineapple Peel Powder):

The pineapple peel powder was oven-dried at 110°C for 24 hours to ensure complete dehydration, then ground using an electric grinder until a fine powder was obtained. Empirical investigations indicated that the most effective solidification ratio was 2% of methyl ethyl ketone peroxide (MEKP), mixed with melted unsaturated polyether at 165°C and 1.5 tons of pressure. This process transformed the mixture into a gel-like substance that hardened at room temperature.

Polymeric composites were synthesized by incorporating pineapple peel powder at weight ratios of 0%, 20%, 30%, and 40% into the unsaturated polyether. The unsaturated polyether liquid was mixed with the pineapple peel powder for 30 minutes to achieve a uniform blend. Subsequently, the solidified additive was added to the mixture, which was then thoroughly combined and poured into specialized molds. The mixture was allowed to cure for 24 hours, forming sheets approximately  $1 \pm 0.1$  mm thick. The samples were carefully extracted from the molds and sectioned into smaller pieces as required for the study.

The weight ratio was determined using the following equation (13):

$$\wp = \frac{W_p}{W_c} * 100 \%$$

$$W_c = W_p + W_m$$

( $\wp$ ) The mass fraction of the powder constituent within the overlay

( $W_c$ ,  $W_m$ ,  $W_p$ ) pineapple peel powder weight, and the base and overlay material, respectively.

In accordance with the standard specifications (ASTM), specific to each individual test. Three samples of peach kernel powder fiber overlays were prepared with unsaturated polyester Table (1):

**Table (1):** Samples of polyesterized unsaturated fortified pineapple peel powder

Samples	No.	Size by micrometer ( $\mu\text{m}$ )	pineapple peel powder mass (gm)	unsaturated polyester mass (gm)
unsaturated polyester	–	130	-	10
unsaturated polyester + pineapple peel powder	1	130	1.0	9.0
unsaturated polyester + pineapple peel powder	2	130	2.0	8.0
unsaturated polyester + pineapple peel powder	3	130	3.0	7.0
unsaturated polyester + pineapple peel powder	4	130	4.0	6.0

### Used Instruments:

#### Thermogravimetric Analysis (TGA) Testing Instrument

Thermogravimetric analysis (TGA) was conducted using a Shimadzu TA-50 instrument, as shown in Figure 1. This device includes a high-precision balance with an oscillating arm and an electric oven, which holds a sample in a platinum cup weighing between 6 and 12 mg. The oven heats the sample in a controlled manner, increasing at a consistent rate of no more than 10°C per minute within a nitrogen atmosphere. The instrument generates graphical data that illustrates the relationship between weight loss and temperature increase. As the temperature rises, the sample weight is continuously monitored until complete decomposition is reached, after which the residual weight is recorded at each temperature interval.



**Figure (1):** Thermogravimetric analysis (TGA and IGT) Shimadzu TA-50

#### **Constant temperature Thermo-weight analyzer (IGA)**

The TGA machine shown in Figure 1 was used; the temperature was set at 350°C before the start of the measurement, and then the model entered and recorded the curves of weight change over time at this temperature.

Thermal treatment of samples:

Five distinct groups of models underwent thermal processing for a duration of 10 hours at the specified temperatures (25, 55, 85, 125, and 155 °C). Upon completion of the designated thermal processing period, the models were removed from the oven and allowed to desiccate under ambient conditions <sup>(14)</sup>.

#### **Results and discussions:**

Thermal stability of polymer composites:

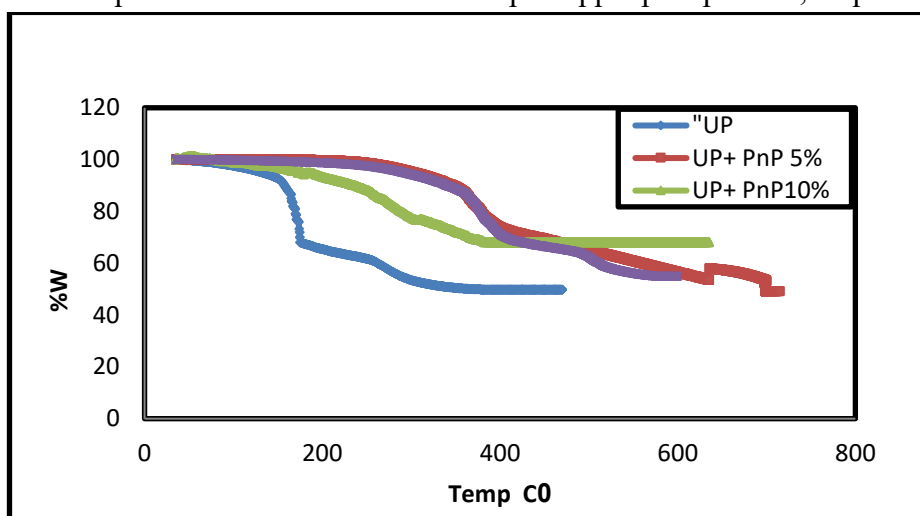
The Thermogravimetric Analysis Instrument (TGA), as shown in Figure 1, was used to study the thermal stability (resistance) of polymer composites, as well as to perform constant-temperature thermogravimetric analysis (TGA). Thermal stability of diverse models was evaluated by contrasting the initial and final temperatures of decomposition, as well as the mass fraction of the residual polymer within the specified temperature range <sup>(15)</sup>:

- **The temperature of the beginning of decomposition (IDT)**
- **Decomposition end temperature (CDT)**
- **The weight ratio at 350°C (Wt%)<sub>350</sub>**

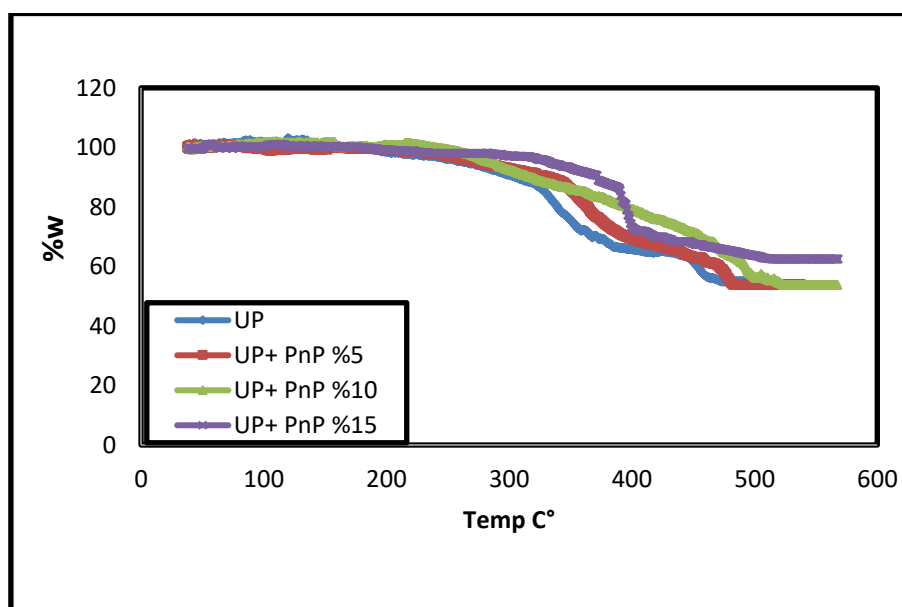
The values of CDT and IDT were determined by the intersection point of the tangents of the TGA at the points of change with the straight part of the curve at the initial and final decomposition. The value of (Wt%)<sub>350</sub> was ascertained by intersecting a vertical line drawn at 350°C with the curve <sup>(16)</sup>.

**Thermal stability assessment of treated aggregates:**

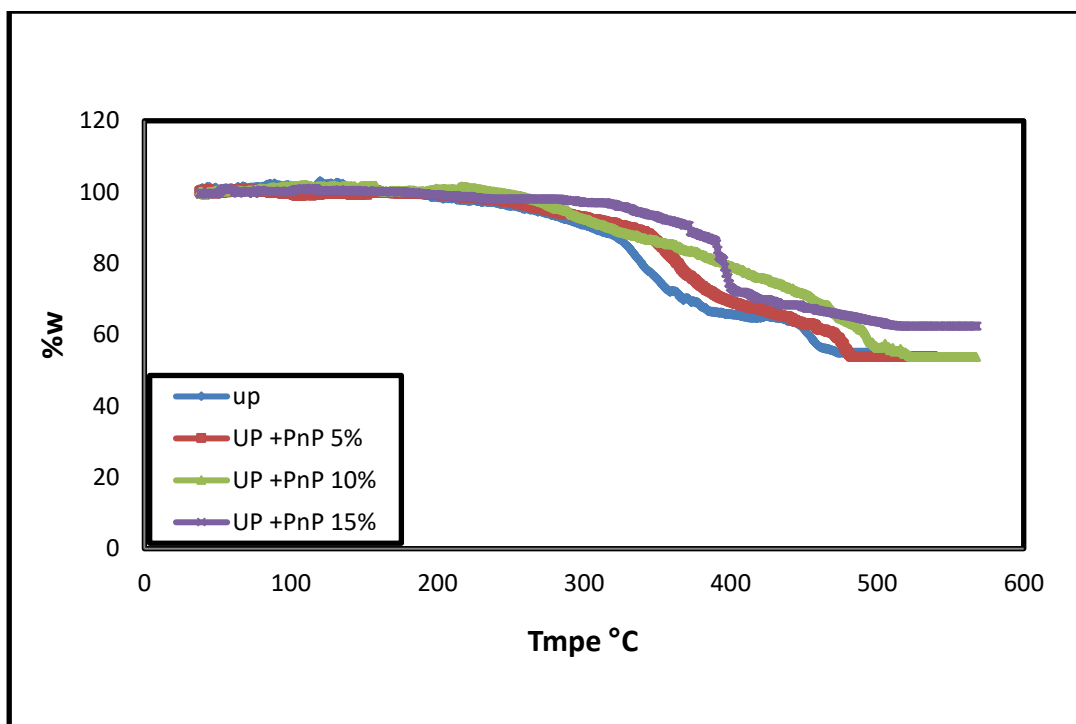
Figures (2-6) indicate the thermo-weight graphical representation of the unsaturated ester and its processed polymer compounds (hardened) at 25, 55, 85, 125, 155 °C, and denote the compositional makeup of the unsaturated ester with pineapple peel powder, respectively.



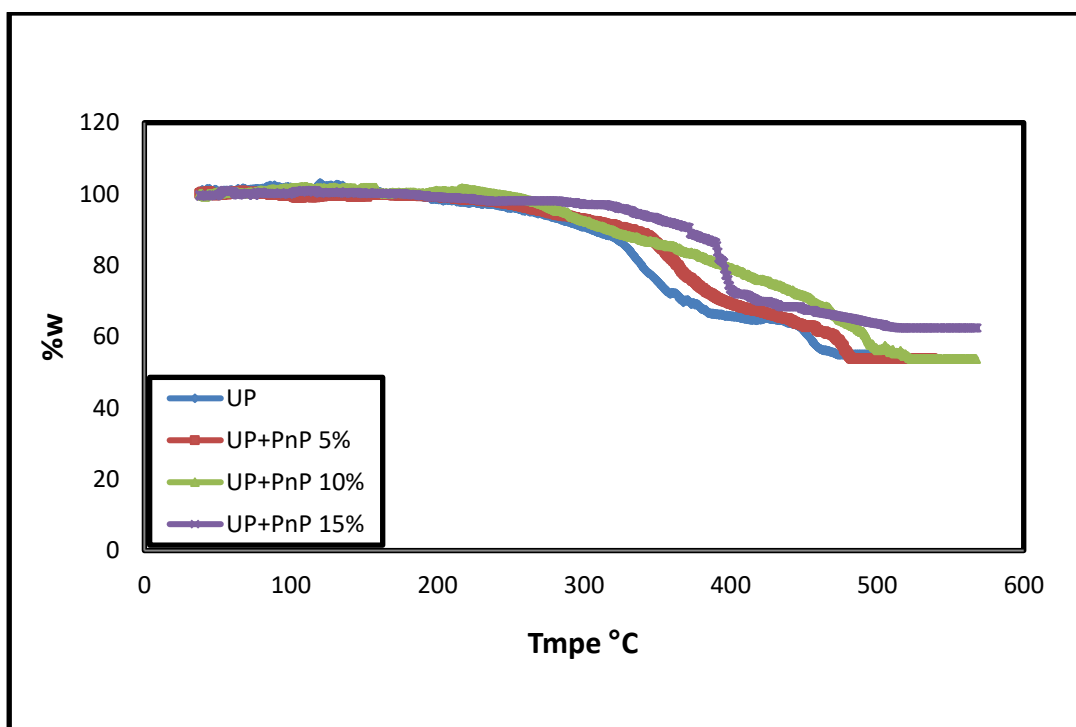
**Figure (2):** Thermo-weight analysis of unsaturated polyesters before and after solidification in different weight ratios at a processing temperature of 25°C



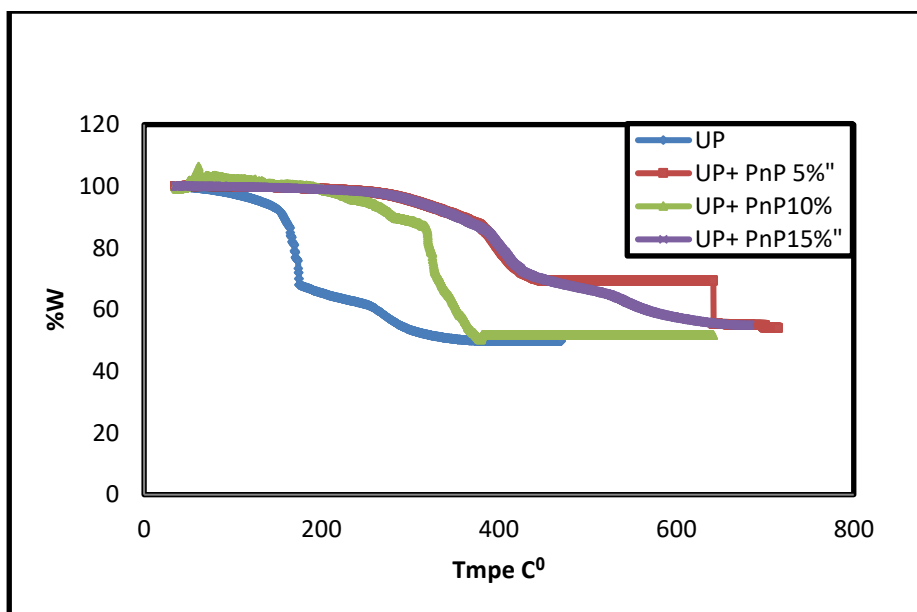
**Figure (3):** Thermo-weight analysis of unsaturated polyesters before and after solidification in different weight ratios at a processing temperature of 55°C.



**Figure (4):** Thermo-weight analysis of unsaturated polyesters before and after solidification in different weight ratios at a processing temperature of 85°C.



**Figure (5):** Thermo-weight analysis of unsaturated polyesters before and after solidification in different weight ratios at a processing temperature of 125°C



**Figure (6):** Thermo-weight analysis of unsaturated polyesters before and after solidification in different weight ratios at a processing temperature of 155°C.

**Table (2):** Thermal stability parameters for the onset of polymer decomposition.

Polymeric overlays	Temperatures of the beginning of decomposition				
	25	55	85	125	155
UPE	180	280	290	310	180
UPE +PnP 5%	420	290	310	300	380
UPE +PnP 10%	210	300	250	280	200
UPE +PnP 15%	390	390	320	290	300

**Table (3):** Thermal stability parameters for the termination of polymer decomposition

Polymeric overlays	Temperatures at the end of decomposition				
	25	55	85	125	155
UPE	440	500	490	490	330
UPE +PnP 5%	720	490	490	495	680
UPE +PnP 10%	400	550	530	530	400
UPE +PnP 15%	590	510	510	500	650

**Table (4):** Mass fraction of residual polymer (w%) at 350 °C for various thermal treatments.

Polymeric overlays	The residual mass of the polymer (w%) at 350 °C				
	25	55	85	125	155
UPE	58	90	75	78	58
UPE +PnP 5%	98	85	80	85	98
UPE +PnP 10%	95	96	90	90	90
UPE +PnP 15%	91	98	98	98	98

By observing Tables (2) and (3) and contrasting the values of (CDT, IDT, and Wt%) for each individual polymeric compound, a noticeable rise in these data is observed due to the presence of the polymer with the unsaturated ester. By extrapolating the IDT values from Table (2), the following findings can be inferred:

1. The incorporation of 15% pineapple peel powder into the polymer composites resulted in significantly elevated IDT values.
2. An inverse correlation was observed between processing temperature and IDT values, indicating that elevated processing temperatures resulted in diminished thermal stability.
3. IDT values for polymer composites containing pineapple peel powder ranged from 180 to 420

Table (3) shows the following effects:

1. The CDT findings of polymer composites containing pineapple peel powder by 15% were high.
2. A tangible effect of increasing the processing heat is observed on the CDT values, since the higher the processing degree, the lower its value.
3. The CDT values for polymer composites containing pineapple peel powder ranged from 330 to 720.

By comparing the mean values of (% Wt) at 350°C with the mean values of both the Initial Decomposition Temperature (IDT) and Complete Decomposition Temperature (CDT), we observe a strong correlation between IDT and CDT values across all solidified unsaturated polyester samples reinforced with pineapple peel powder.(17-16)

Experimental results indicate that thermal processing conditions have a significant impact on the IDT and CDT values of polymer composites. Specifically, these values show a negative correlation with increased processing temperatures and weight ratios. This behavior suggests that, in polymer overlays, the polymer chains overlap, creating a cohesive structure. As the temperature rises, the polymer chains become more fragile and disintegrate faster due to inherent softening and the increased rotational movement of their constituent units (18-19). The consistently high CDT values highlight the material's strong thermal stability.(21-20)

Additionally, an inverse correlation was observed between processing temperatures and IDT values of CDT, as well as the percentage weight (wt%) at 350°C, for polymer composites treated

at elevated temperatures ranging from 25 to 155°C. As processing temperatures increased, these values declined, suggesting easier separation and movement among the polymer's units.(23-22)

The results clearly demonstrate a distinction between the lowest and highest processing temperatures (25°C and 155°C, respectively). This phenomenon can be attributed to the role of unsaturated polyester compounds, which stabilize the second polymer and prevent its dissociation at relatively low temperatures, whereas the material partially dissociates when isolated (24-25).

## References:

1. Pec,as P, Carvalho H, Salman H, et al., (2018),” Natural fibre composites and their applications”, a review. J Compos Sci; 2(4): 66.
2. Ebtehaq Z. Sulyman, Noaman Z. Sulyman, (2020), "Preparation of Polymeric Composites from Polypropylene and Palm Fronds and the Study of Some of Their Physical Properties", Baghdad Science Journal, 17(3):772-779.
3. Rashid, Abdul Rahman Rashid Muhammad, (2019), “A study of the effect of direction, volume fractions and temperature on the flexural strength property of polymer sheets reinforced with glass fibers”, (Master’s thesis, Al-Qadisiyah University).
4. Anwar M. Ahmed, Ebtehaq Z. Sulyman, (2022),” Preparation and study of mechanical and thermal properties of polymeric composites of unsaturated and polyester cellulose fibers”, International Journal of Health Sciences, 6(S1), 9305–9321.
5. Anwar M. Ahmed, & Ebtehaq Z. Sulyman (2023), "Improving Mechanical and Thermal Properties for Unsaturated Polyester Resin by Adding Sisal Fibers" College of Basic Education Researchers Journal. 19(1) ,7452-1992.
6. Esraa S. Waad Alla, Ebtehaq Z. Sulyman, (2021),” Preparation of polymeric Composites From Saturated Polyesters Glass Powder (Flourscent) and Study of Its Mechanical Properties Preparation of polymeric Composites From”,Iraqi Journal of Market Research and Consumer Protection,13(1),131-139.
7. Farah S, Anderson DG and Langer R., (2016), —Physical and mechanical properties of PLA, and their functions in widespread applications|- a comprehensive review. Adv Drug Deliver Rev, 107(1), 367–392.
8. Nathan Schwarz, Hieu Cam, Narayanan Neithalath, (2008),” Influence of a fine glass powder on the durability characteristics of concrete and its comparison to fly ash, Cement & Concrete Composites”, 30, 486–496.
9. Chikhalikar S.M. and Tande S.N. (2012), “An Experimental Investigation on Characteristics Properties of Fibre Reinforced Concrete Containing Waste Glass Powder as Pozzolona”, 37th Conference on Our World in Concrete and Structures, Singapore, August.
10. Ebtehaq Z. Sulyman, Amel G. Abed, Abeer Q. Younis, (2019),” Gravimetric and differential thermal analysis and activation energy Studies of unsaturated polyester using date seeds powder and walnutShells”, International Journal of Research in Pharmaceutical Sciences, 10(4), 3811-3821.

11. Maysam, K. gasem and Ebtehag, Z., Sulyman, (2022). Thermal and mechanical study of polymeric composites prepared from epoxy with iron filings. *International Journal of Health Sciences*, 6(S4), 9708–9719.
12. Ebtehag, Z., Sulyman, H.A. Salih, T. A. Hallow, " Preparation of a polymeric aggregate of unsaturated Poly ester with sawdust and studying its physical and mechanical properties", *Int. J. Res. Pharm. Sci.*, 10(3), 2280-2289 (2019).
13. Ebtehag, Z., Sulyman,, A.G. Abed, A. Q. Younis, Gravimetric and differential thermal analysis and activation energy Studies of unsaturated polyester using date seeds powder and walnut shells, *Int. J. Res. Pharm. Sci.*, 10(4), 3811-3821 (2019).
14. Ebtehag, Z., Sulyman, N. Z. Sulyman, "Preparation of Polymeric Composites from Polypropylene and Palm Fronds and the Study of Some of Their Physical Properties", *Baghdad Science Journal*, 17(3):772-779 (2020).
15. R. E. Ibrahim, Ebtehag, Z., Sulyman, Preparation Polymeric Composites from Epoxy with Randomly Woven Fiber Glass and Studies the Mechanical Properties, *Rafidain Journal of Science*, 28(3), 105-115(2019).
16. Ebtehag, Z., Sulyman, and A. A. Mohamed, Thermal Gravimetric Analysis of Physical Networks of Epoxy resin with N-2-Methyl Phenyl Maleamic acid, *Proceedings of the second scientific conference*, (College of Science, University of Mosul, 2011 Nov.).
17. N. Z. Sulyman, Ebtehag, Z., Sulyman, T. A. Hallow, " Study the Thermal Properties and Effect of Acidic Solutions to Unsaturated Polyester Composite with Iron Filing", *Systematic Reviews in Pharmacy*, 12(1), PP.1199-1205(2021).
18. R. E. Ibrahim, Study of Some Mechanical and Physical Properties for Epoxy Composite Reinforced with Glass Fiber and Carbon Fiber, M.Sc. Thesis, Department of Chemistry, College of Education for Girls, University of Mosul, 2019.
19. Dalia, R. Saadallah , Ebtehag Z. Sulyman, (2022) . Preparation and study of some mechanical and thermal properties of epoxy composites with natural styrene butadiene rubber (damaged tires). *International Journal of Health Sciences*, (I), 6278-6291.
20. Dalia, R. Saadallah, Ebtehag Z. Sulyman, (2022). Preparation and study of mechanical and thermal properties of an epoxy composite with isobutyl rubber: Damaged water taps powder and signals. *International Journal of Health Sciences*, (II), 5200-5210.
21. Esraa S. Waad Alla, Ebtehag Z. Sulyman, (2021),” Preparation of polymeric Composites from Saturated Polyesters Glass Powder (Flourscent) and Study of Its Mechanical Properties Preparation of polymeric Composites From”, *Iraqi Journal of Market Research and Consumer Protection*, 13(1), 131-139.
22. Esraa S. Waad Alla, Ebtehag Z. Sulyman, (2021),” Preparation of polymeric Composites from Saturated Polyesters Glass Powder (Flourscent) and Study of Its Mechanical Properties Preparation of polymeric Composites From”, *Iraqi Journal of Market Research and Consumer Protection*, 13(1), 131-139.
23. Maysam, K. gasem and Ebtehag, Z., Sulyman, (2022). Thermal and mechanical study of polymeric composites prepared from epoxy with iron filings. *International Journal of Health Sciences*, 6(S4), 9708–9719.
24. Safa, D, Mohmmmed, Ebtahag, Z, Sulyman, Huda, A, Abid, (2021),” Study the mechanical and physical properties of an epoxy compound with silicate sodium”, *Iraqi Natinol Journal of chemistry* , 21(1), 8-18.

25. Safa, D, Mohmmmed, Ebtahag, Z, Sulyman, Rand Saad-Aldeen, & Eklas, Q, Hammadi, (2021). Study the mechanical and physical properties of an epoxy compound with Sulfur. *Iraqi National Journal of Chemistry*, 21(3), 22-31.