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## Studying the Preparation and Characterization of Composite Polyester Using Different Additives

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**Abstract:** Polyester is defined as one ester as a minimum or by using an additional linking group for separately repeating a unit polymer. Polyesters are classified into two types. The first is thermoplastic polyester, while the second is unsaturated polyester (UP) cured with highly cross-linked thermosets. UP is defined as a polyester that emerges from condensation. Due to the difficulty of handling UP and its brittleness at room temperature, UP is sold as such. As an alternative, in the molten state, UP is mixed with the vinyl monomer every time it is synthesized freshly in a chemical plant. We will focus on studying UP and its resins in this manuscript. Monomers, vinyl monomers, synthesis, manufacture, special additives, fillers, reinforcing materials, curing, mechanical properties, and applications for UP will be discussed thoroughly in this research. The six most essential fillers used in this research (e.g., calcium carbonate, wood flour, nano silicon oxide, sand, shredded automobile tires, and glass fiber) were considered to minimize the cost of cured materials and improve their mechanical qualities.

**Keywords:** polyester, additive, fillers.

### 不同助剂复合聚酯的制备及表征研究

**摘要:** 聚酯被定义为至少一种酯或通过使用额外的连接基团来单独重复单元聚合物。聚酯分为两类。第一种是热塑性聚酯, 第二种是用高度交联的热固性树脂固化的不饱和聚酯(向上)。向上被定义为从缩合中产生的聚酯。由于向上难以处理且在室温下易碎, 因此向上以这种形式出售。作为替代方案, 在熔融状态下, 向上每次在化工厂新合成时都会与乙烯基单体混合。在这份手稿中, 我们将重点研究向上及其树脂。本研究将深入讨论向上的单体、乙烯基单体、合成、制造、特殊添加剂、填料、增强材料、固化、机械性能和应用。本研究使用的六种最基本的填料(例如碳酸钙、木粉、纳米氧化硅、沙子、碎汽车轮胎和玻璃纤维)被认为可以最大限度地降低固化材料的成本并提高其机械质量。

**关键词:** 聚酯、添加剂、填料。

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## 1. Problem Statement

The polymer industry has grown to play a critical role in producing everyday products. For example, unsaturated polyester (UP) is a thermoset polymer known for its essential mechanical and physical qualities, leading to increased industrial use in recent years. Also, many kinds of primary materials can be used, so a wide range of properties can be adjusted. Unsaturated polyester is employed as a pure resin form, with or without additives or reinforcements [1]. Polyester resins are used for polyester concrete, coatings, electronic and microwave industries, bone cement, road paints, toner materials, reactive melt modifiers, and compatibilizers [2]. Although their manufacturing is growing, their applications in ornamental arts are not widely known. They can be done modestly with the correct mixing ratios, fillers, and reinforcing materials [3, 4]. Therefore, we did some tests by adding fillers and reinforcing materials with different percentages to form or achieve a new composite material characterized by unique properties with significant resistance or impact.

## 2. Research Objective

The main objective of this research was to discuss and analyze monomers, vinyl monomers, synthesis, manufacture, special additives, fillers, reinforcing materials, curing, mechanical properties, and applications for UP. We also discuss the preparation and characterization of a composite linear UP, called general purpose, without any filler. Then, many fillers with different percentages in the composite linear UP preparation (e.g., calcium carbonate, wood flour, nano silicon oxide, sand, shredded automobile tires, and glass fiber) were considered. During this process, new composite materials characterized by new properties with significant resistance or impact are expected to be formed or achieved.

## 3. Research Methods

Experimental work included the preparation of a composite linear UP called general purpose without any addition of filler (pure sample). For this, 42% monopropylene glycol, 42.2% phthalic anhydride, 15.7% maleic anhydride, 0.04% stabilizer antioxidant, and a ratio of 100:55 polyesters to styrenes were used. It was dissolved in a styrene monomer. Using benzoyl peroxide as an initiator and cobalt octanoate as an activator, the preparation of composite linear UP also included the addition of many fillers. These fillers were used in different percentages to obtain samples with different properties. Then, mechanical tests, such as tensile,

compression, and impact tests, were used to measure and compare the mechanical properties of each sample.

## 4. Literature Review

### 4.1. UP and UP Resin Backgrounds

Along with the short polyester chain, which includes both polymerizable double bonds and a vinyl monomer, we conclude that UP resins consist of two polymers. The curing reaction consisted of vinyl monomer copolymerization with the polyester double bond. A three-dimensional network was produced or formed during the curing course. UP resins belonged to the thermosets group by comparing UP to UP resins. UP is defined as polyesters that emerge at the condensation level [5, 6]. Due to its difficulty being handled besides its brittle at room temperature, it sold as such. As an alternative, in the molten state, it is mixed with the vinyl monomer every time it is freshly synthesized in a plant [7]. The materials characterized as viscous at room temperature, with around 60% styrene content, were sold. The mixture of vinyl polymer with UP is referred to as UP resin [8]. UP resins are products of linear polycondensation that depend on saturated and unsaturated acids to both anhydrides and diols [9]. Also, oxides may be included. These resins are characterized by low-degree polymerization pale yellow oligomers [10, 11]. According to molecular weight and chemical composition, we can conclude whether these oligomers are viscous liquids or brittle solids (1200–3000 g/mol) with a range of 200–2000 cps viscosity of the obtained diluted unsaturated resin solution. Polyester resins are widely used worldwide due to their significant versatility and low cost [12-20].

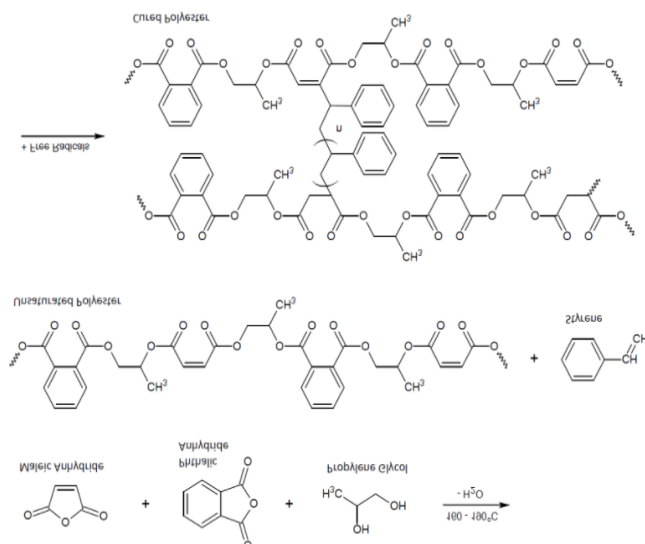


Fig. 1 Unsaturated polyester reaction

#### 4.1.1. Fillers Used in Polyester

A wide range of fillers was available for use. We mention some examples, such as calcium carbonate powder, barium sulfate, clay, cellulose yarn, talc, silica sand powder, glass powder, silica powder, glass beads, alumina powder, mica, river sand, crushed stone, aluminum hydroxide, white marble, and marble scrap, among others. For barium sulfate and glass powder, and aluminum hydroxide cases, translucency was imparted during the curing process. The following table includes the most commonly used fillers.

Table 1 List of common fillers

Filler
Bentonite
Calcium carbonate
Clay
Glass beads
Flyash
Woodflour
Rubber particles
Nanocomposites

The advantages of fillers included reducing costs and enhancing the mechanical characteristics of the cured materials [21, 22]. Bentonite, montmorillonite, and fly ash were all considered organic fillers. We used six fillers in this study, including calcium carbonate, wood flour, nano silicon oxide, sand, shredded automobile tires, and glass fiber.

#### 4.2. The Curing of UP Resin Reaction

For polyester chains, as well as styrene and possibly other monomers, the curing of the UP takes place by free polymerisation radical via unsaturated groups (double bonds), as the following figure shows schematically. The free radicals are obtained through the use of peroxide, as it can be decomposed and it is defined as the free radicals rate in order to be generated. Consequently, it has the ability to govern both resin gel and cure time [23-25].

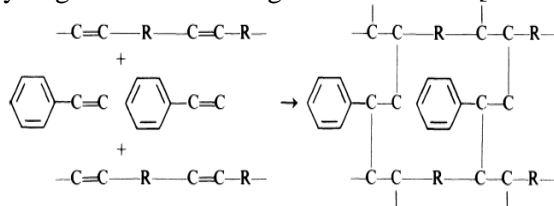


Fig. 2 UP resins reaction

Initiators that can be used include peroxides and azo and azine compounds; there is a wide range of initiators depending on the curing T [26, 51-53]. In case of large hand lay-up structures at room T, MEKP (the commercial name of methyl ethyl ketone), was used. Benzoyl peroxide, on the other hand, was selected for curing at moderate T (~60°C–90°C). Finally, di-t-butyl was employed for ~130°C–150°C), which is considered

as hot press; t-butyl perbenzoate may also be included to be used as hot press. However, when a large T increase is expected, a mixture of initiators is required [27, 46-50]. Some metal oxides, in addition to tertiary amines and mercaptans, may be employed to accelerate the decomposition of peroxides [28, 41-45]. Naphthenate (C<sub>22</sub>H<sub>14</sub>CoO<sub>4</sub>) and octanoate ((C<sub>7</sub>H<sub>15</sub>COO)<sub>2</sub>Co) cobalt are the most frequently employed accelerators [29]. The process consists of three stages, and the following are the steps involved in the curing of polyester resins: the first stage is known as gelation in which resins are converted from a free-flowing liquid into a soft gel; the second stage is known as hardening, whereby resins are converted from a soft gel into a solid material so that they can be easily removed from the mould; the third stage includes final curing, as the resins are considered to be able to reach the desired chemical and mechanical properties; this may take many hours at elevated T [30-35].

## 5. Experimental Work

### 5.1. Polyester

The procedures for adding the fillers to the linear UP include [36-40]:

- Preparing a clean and dry 250 ml beaker;
- Weighing the empty 250 ml beaker on a balance and taking the readings;
- Placing about 120 g of the linear UP solution into the empty 250 ml beaker;
- Adding the 0.24 g cobalt octoate activator with as activators are added at 0.2%;
- Stirring the cobalt octoate activator with the linear UP solution for about five minutes and, as if we added the initiator directly, a great explosion will take place;
- Adding 1.2 g of benzoyl peroxide as the initiators should be added at 1%;
- Adding the fillers with different percentages;
- Stirring the mixture for five minutes;
- Leaving the mixture for two minutes before pouring, in order to avoid bubbles;
- Pouring the mixture into one Petri dish and two test tubes;
- Leaving the prepared composite for about two to four hours, in order to reach completed curing and hardening;
- Break both test tubes and Petri dishes, in order to take the sample out;
- Usually we have pure cross-linked polyester without any addition of fillers, in order to compare it to the sample with filler;

- Measure the mechanical characteristics of the compression, tension, and impact tests to get the plots and results.

## 6. Results

### 6.1. Mechanical Tests of Polyester

Table 2 Tensile test

Sample	Load (KN)	Time (S)	Elongation (%)	Stress (MPa)	Strain (%)
Pure	6.260	27.753	8.8730	40.649	17.746

Table 3 Compression test

Sample	Load (KN)	Time (S)	Elongation (%)	Stress (MPa)	Strain (%)
Pure	6.49	21.259	6.6990	64.900	13.398

#### 6.1.1. Impact Test

$E = mgh$  (where  $m$  is the ball's mass and  $h$  is the ball's fall height)  $g = 9.81 \text{ N}$

$$E = 1.4 \text{ Kg} * 9.81 \text{ N} * 0.16 \text{ m}$$

$$E = 2.19744 \text{ Joule}$$

### 6.2. Mechanical Tests of Shredded Automobile Tires

Table 4 Tensile test

Sample	Load (KN)	Time(S)	Elongation (%)	Stress (MPa)	Strain (%)
Pure	6.260	27.753	8.873	40.649	17.746
0.5%	4.875	29.452	9.440	31.656	18.880
1%	4.515	28.321	8.620	29.318	16.640
1.5%	4.365	26.654	8.480	28.344	16.960
2%	4.100	19.261	6.036	26.623	12.072
2.5%	3.275	17.463	5.420	21.266	10.840
3%	3.185	17.243	5.380	20.682	10.924

Shredded automobile tires

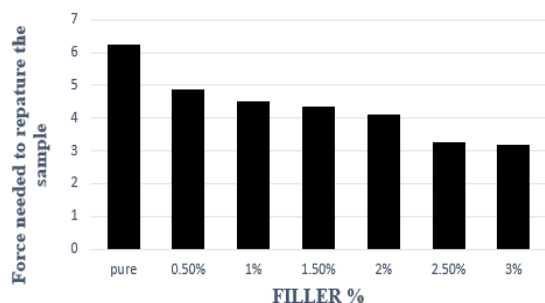


Fig. 3 Tensile test

Table 5 Compression test

Sample	Load (KN)	Time (S)	Elongation (%)	Stress (MPa)	Strain (%)
Pure	6.49	21.259	6.699	64.900	13.398
0.5%	7.20	20.36	6.406	72	12.812
1%	6.91	20.24	6.387	69.50	12.174
1.5%	6.39	18.463	6.285	63.46	11.494
2%	6.17	18.162	6.100	61	11.248
2.5%	5.69	16.864	5.238	36.9	10.476
3%	5.49	15.961	5.100	31.429	9.836

Shredded automobile tires

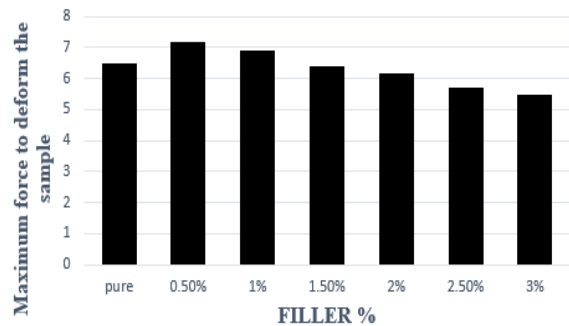


Fig. 4 Compression test

Table 6 Impact test

Sample	Energy (Joule)
Pure	2.19744
0.5%	3.15882
1%	4.8069
1.5%	6.1803
2%	2.7468
2.5%	2.0601
3%	1.64808

Shredded automobile tires

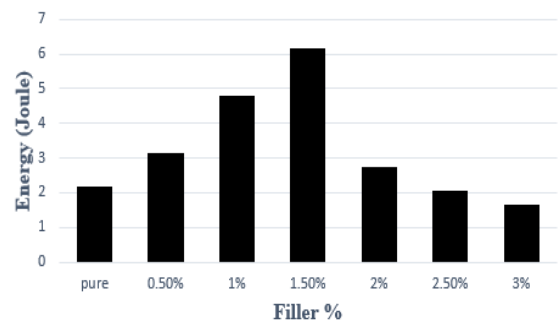


Fig. 5 Impact test

### 6.3. Mechanical Tests of Sand

Table 7 Tensile test

Sample	Load (KN)	Time (S)	Elongation (%)	Stress (MPa)	Strain (%)
Pure	6.260	27.753	8.873	40.649	17.746
5%	4.425	24.757	7.8560	28.734	15.712
10%	4.295	22.858	7.2170	27.890	14.434
15%	4.150	21.758	6.8480	26.948	13.696
20%	3.815	19.362	6.0470	24.773	12.094
25%	3.650	18.575	5.8700	23.701	14.606
30%	3.525	17.263	5.3690	23.539	10.738

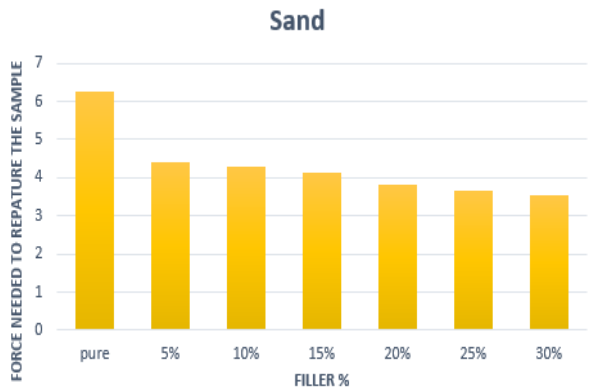


Fig. 6 Tensile test

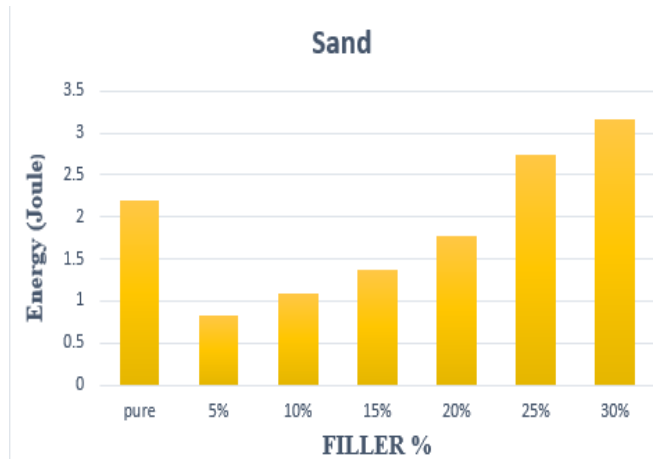


Fig. 8 Impact test

Table 8 Compression test

Sample	Load (KN)	Time (S)	Elongation (%)	Stress (MPa)	Strain (%)
Pure	6.49	21.259	6.699	64.900	13.398
5%	5.11	14.566	4.4720	33.182	8.944
10%	6.07	15.390	4.7320	60.750	11.172
15%	6.22	16.064	4.9460	62.150	9.892
20%	6.58	16.479	5.3120	65.850	15.480
25%	6.72	17.464	5.4290	67.150	10.858
30%	7.10	18.463	5.7630	76.104	11.526

### 6.4. Mechanical Tests of Calcium Carbonate

Table 10 Tensile test

Sample	Load (KN)	Time (S)	Elongation %	Stress (MPa)	Strain (%)
Pure	6.260	27.753	8.873	40.649	17.746
5%	4.635	25.756	8.1650	30.097	16.330
10%	4.425	20.361	6.4020	28.701	12.804
15%	3.790	22.558	7.200	24.610	14.66
20%	3.780	21.860	6.8680	24.545	13.736
25%	3.205	18.263	5.6850	20.812	11.370

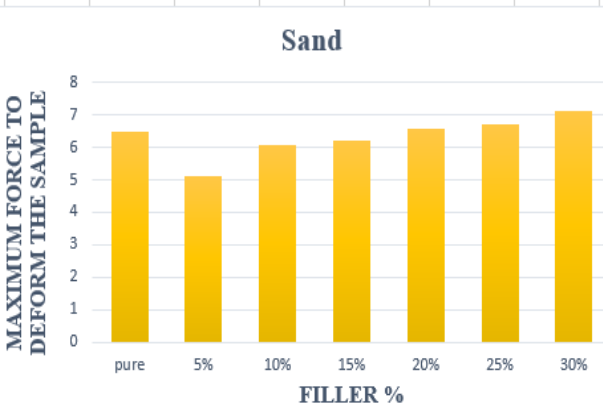


Fig. 7 Compression test

Table 9 Impact test

Sample	Energy (Joule)
Pure	2.19744
5%	0.82404
10%	1.09872
15%	1.372
20%	1.7836
25%	2.744
30%	3.1556

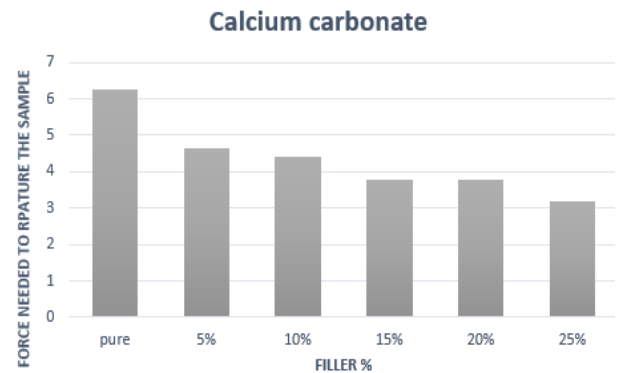


Fig. 9 Tensile test

Table 11 Compression test

Sample	Load (KN)	Time (S)	Elongation %	Stress (MPa)	Strain (%)
Pure	6.49	21.259	6.699	64.900	13.398
5%	4.14	17.464	5.4350	26.883	10.870
10%	4.41	18.563	5.7940	28.571	11.588
15%	4.87	19.362	6.0650	31.623	12.130
20%	6.95	26.953	8.5990	44.968	17.198
25%	7.35	28.652	9.1640	45.065	18.328

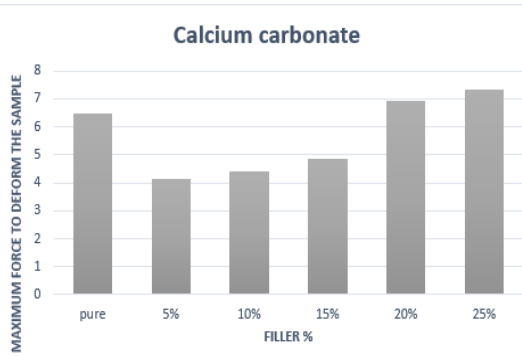


Fig. 10 Compression test

Table 12 Impact test

Sample	Energy (Joule)
Pure	2.19744
5%	1.3734
10%	2.7468
15%	3.4335
20%	4.8069
25%	6.1803

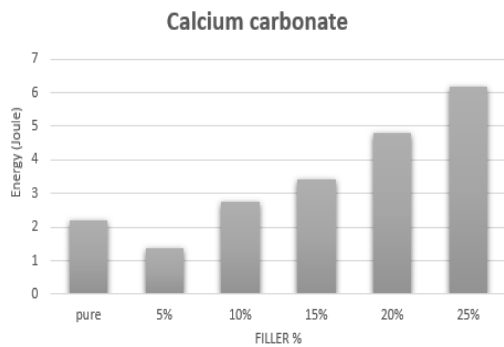


Fig. 11 Impact test

### 6.5. Mechanical Tests of Silica

Table 13 Tensile test

Sample	Load (KN)	Time (S)	Elongation %	Stress (MPa)	Strain (%)
Pure	6.260	27.753	8.873	40.649	17.746
0.5%	1.990	11.369	3.3900	12.922	6.780
1%	1.930	9.370	2.7310	12.532	5.462
1.5%	1.680	8.072	2.2840	10.909	4.568
2%	1.345	8.972	2.5900	8.734	5.180
2.5%	1.100	5.775	1.5190	7.143	3.038

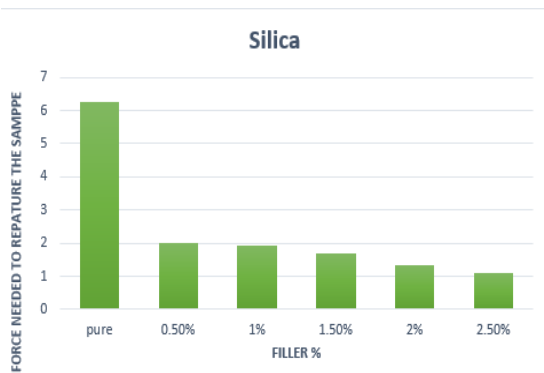


Fig. 12 Tensile test

Table 14 Compression test

Sample	Load (KN)	Time (S)	Elongation %	Stress (MPa)	Strain (%)
Pure	6.49	21.259	6.699	64.900	13.398
0.5%	7.42	24.956	7.9200	48.182	15.840
1%	5.71	17.463	5.4110	44.100	10.822
1.5%	5.68	117.457	5.3830	36.883	10.654
2%	4.13	16.340	5.1000	34.800	10.051
2.5%	2.26	8.272	2.3570	22.550	4.714

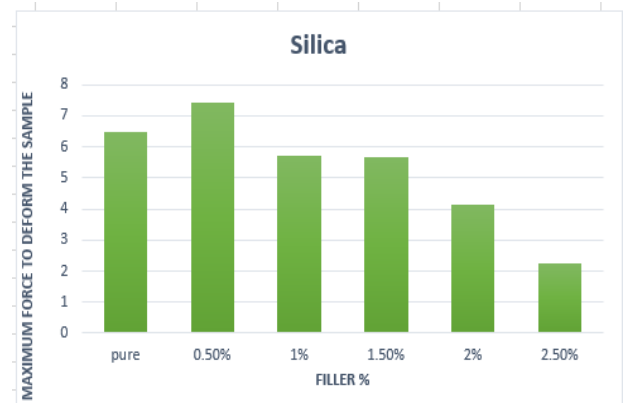


Fig. 13 Compression test

Table 15 Impact test

Sample	Energy (Joule)
Pure	2.19744
0.5%	0.82404
1%	1.23606
1.5%	2.0601
2%	3.29616
2.5%	4.1202

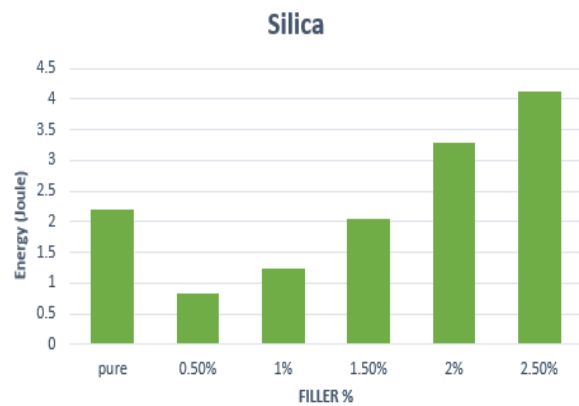


Fig. 14 Impact test

### 6.6. Mechanical Tests of Glass Fiber

Table 16 Tensile test

Sample	Load (KN)	Time (S)	Elongation %	Stress (MPa)	Strain (%)
Pure	6.260	27.753	8.873	40.649	17.746
0.10%	5.815	23.257	7.3370	37.760	14.674
0.15%	4.380	18.862	5.8920	28.442	11.784
0.25%	4.315	17.363	5.4030	28.019	10.806
0.35%	4.195	16.264	5.0460	27.110	10.492
0.45%	4.025	15.156	4.6690	26.136	9.338

### 6.7. Mechanical Tests of Wood Flour

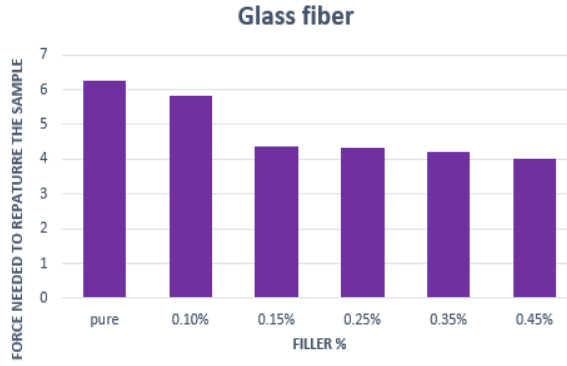


Fig. 15 Tensile test

Table 17 Compression test

Sample	Load (KN)	Time (S)	Elongation (%)	Stress (MPa)	Strain (%)
Pure	6.49	21.259	6.699	64.900	13.398
0.10%	9.32	25.256	8.0230	60.552	16.046
0.15%	8.98	22.262	7.6920	58.800	14.384
0.25%	6.16	19.661	6.1510	50.800	12.302
0.35%	3.39	16.463	4.995	20.500	9.990
0.45%	2.94	15.165	4.5630	15.700	9.126

Table 19 Tensile test

Sample	Load (KN)	Time (S)	Elongation (%)	Stress (MPa)	Strain (%)
Pure	6.260	27.753	8.873	40.649	17.746
1%	3.565	23.257	7.3430	23.149	14.686
1.5%	3.372	21.559	6.7760	21.916	13.552
2.5%	3.265	16.664	5.1550	21.201	10.310
3%	3.075	15.730	5.0120	19.968	11.428
4%	2.645	15.365	4.970	17.175	10.154
5%	2.330	14.067	4.2790	15.130	8.558

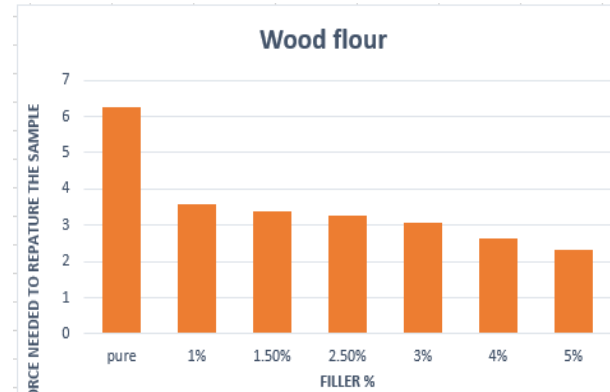


Fig. 18 Tensile test

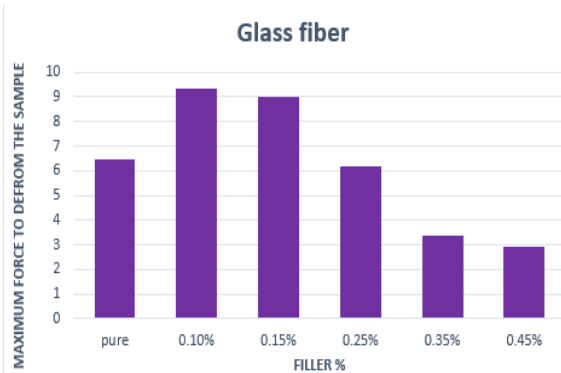


Fig. 16 Compression test

Table 20 Compression test

Sample	Load (KN)	Time (S)	Elongation (%)	Stress (MPa)	Strain (%)
Pure	6.49	21.259	6.699	64.900	13.398
1%	7.05	23.059	7.2870	70.500	14.574
1.5%	6.02	19.962	6.2460	60.200	12.492
2.5%	5.61	19.661	6.1500	56.100	12.300
3%	5.42	19.561	6.1250	57.383	12.220
4%	4.80	17.663	5.4790	48	10.958
5%	4.60	15.865	4.600	46	9.754

Table 18 Impact test

Sample	Energy (Joule)
Pure	2.19744
0.1%	2.47212
0.15%	2.7468
0.25%	3.57084
0.35%	4.39488
0.45%	5.08158



Fig. 19 Compression test

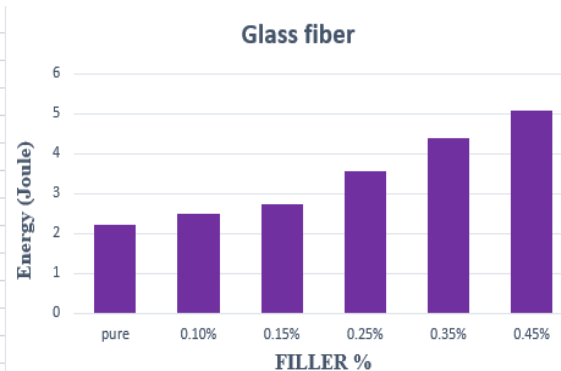


Fig. 17 Impact test

Table 21 Impact test

Sample	Energy (Joule)
Pure	2.19744
1%	1.92276
1.5%	2.47212
2.5%	2.88414
3%	3.57084
4%	3.84552
5%	4.39488



Fig. 20 Impact test

## 7. Conclusion

There were numerous stages to the research. First, to investigate the polyester resin's curing period, as well as studying and choosing the best polyester according to the percentages of both the accelerator and the initiator. We can conclude that the range of the curing time was about 35 minutes up to 50 minutes, the accelerator weight was equal to 0.2%, and the initiator weight was equal to 1%. The second stage included the preparation of composite linear UP with the addition of many fillers with different percentages, such as calcium carbonate, wood flour, nano silicon oxide, sand, shredded automobile tires, and glass fiber. This was conducted to form or achieve a new, composite material characterized by new properties that showed great resistance or impact. We can conclude that this showed a great resistance to the applied energy with the impact test in all cases. The addition of shredded automobile tires had the highest value recorded, at 6.1803 joule and with sample of 25 % calcium carbonate. According to all tensile results of all fillers, we can conclude that the maximum load that must be applied to a sample in order for it to be broken down was displayed without the use of any filler. The sample indicates its strength by increasing the applied force, as breaking it down demands a high value or loading force. But with the addition of a glass fiber filler, we reached the same value as with the pure sample, so glass fiber is considered to be the best filler. According to all compression results, we can conclude that there are about 80% of fillers that exceed many pure sample values, but the best value and performance was recorded with the addition of 0.1% glass fiber, which is equal to 9.32 kN.

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