Study of the mechanical and physical properties of bio-composite material based on wheat starch and wheat straw fibers

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Abstract. Natural fiber reinforced bio-composites are taking a big role in various sections around the world such as industry, construction, and health section. In this study, a bio-composite material based on wheat starch matrix and natural wheat fibers is prepared. Five different specimens with following fiber contents 50, 54.5, 58.3, 61.5, and 64.2% by weight are added to wheat starch matrix. Mechanical and physical tests are performed for different fiber weight contents. Charpy impact test results showed that impact energy is decreasing with increase in fiber content. Three-point deflection test data show that as higher fiber content as higher flexural stress is obtained. Shore D hardness test results show that the hardness values increase as fiber content increase. Water uptake test results showed that water absorption also decreases by increasing fiber content. An optical investigation is performed to calculate the average length and width of chopped fibers used as reinforcement. The average length and width of fibers were found to be 5.73mm and the average width is 0.38mm.

1. Introduction
The environmental limits and new rules on recycling of hazardous and waste materials have pushed manufacturers to develop revolutionary materials from sustainable sources. The use of natural fillers in polymer matrix composites can introduce an important advantages compared to regular filling materials used in composite materials like carbon-glass fibers [1]. Polymer-based natural resources fiber composites have become more popular around the world because of their affordability, low density, sustainability, biodegradation character, and appropriate mechanical properties [2] also due to these materials being environmentally friendly [3]. Many research articles [4–9] have worked on composite materials to get advantage of the great and diverse properties of filling materials like unique thermal and mechanical properties [10,11]. These researches focus primarily on variable filler content as well as their chemical composition and polymer matrix selection.

The mechanical characteristics of particle-reinforced composites are heavily dependent on many factors like: composite material component distribution, component adhesion, morphology and size of particles. It is found that particles have a clear impact on these mechanical characteristics [12]. For instance, smaller particles of calcium carbonate provide a higher strength values for filled composites of polypropylene at a specific load [9]. The hydrophilic property of natural fillers, however, results in poor compatibility with the hydrophobic polymer based matrix and bad dimensional constancy as water absorption increase the volume of fillers [13]. Modification of polymers improves the bonding forces between particles when one end of the molecule is attached to the reinforced surface, whereas the other end bonds with the polymer phase. The most commonly used chemical treatments and polymer modification of fillers are to ensure excellent bonding force and boost the impact resistance.
and elongation capability of the composite [14]. Different natural fibers like coir [16], Alfa [15], banana [17], clay [19], calcium carbonate [18], and graphene [20] have been investigated as innovative reinforcement factors in polymeric composite materials. The growing interest in natural fiber-reinforced composites led many researchers around the world to conduct many studies in the direction of preparing and characterizing new materials. Chandramohan, D., & Kumar, A. J. [21] presented an experimental research on the preparation of polymer-based composites. Different natural fibers like walnut and powdered coconut shell, and rice husk are used as reinforcement materials with natural epoxy resin to form composite samples. The researchers have used a fiber composition in each specimen 1:1 while the hardener to resin ratio was 1:10. The synthesized composites were tested according to ASTM standards to calculate the mechanical properties like tensile, shear, flexural, and impact strengths. These tests evaluated the fore mentioned properties in both dry and wet samples. Test results show that under mechanical loads, hybrid composites have much better mechanical properties than single fiberglass reinforced composite. However, the results of research reveals that the mixture of walnut and coconut shell fibers can enhance the mechanical characters. Shubhra et al. [22] have used a compression molding technique to prepare a silk reinforced gelatin-based composites. The composite fiber content was 20% by weight. Mechanical properties like tensile strength and modulus, bending strength and modulus, hardness value and impact strength were found as: 44.5 MPa, 0.65 GPa, 63 MPa, 3.7 GPa, 96 hardness, and 5.1 kJ/m² respectively. The study also addresses the environmental degradation of composite by simulating a 30 hours weathering test. The results of this sections showed a loss of 15.2% of the tensile strength by the end of the weathering test.

The study included a biodegradation investigation showing that the material under study has degraded in a fast manner resulting a 52.1% loss of weight at the end of a 24 h test. A structure analysis was also performed to visualize the fiber pull-out and fracture of the samples. Sature, P., & Mache, A. [23] made a study concentrating to examine the mechanical characteristic and act of combined jute/hemp epoxy composites as compared to pure jute and hemp fiber-reinforced composites. The major disadvantage of natural fiber-based composite materials is the hydrophilic nature which is the reason for the tendency to absorb wetness. So as to protect the natural fiber-based composites from this disadvantage, researchers placed a sheet of woven glass carpet as the outer cover one on each side of the hybrid laminate of jute-hemp. Mechanical properties like tensile, compression, shear and flexural strength were gained along with the water immersion test. From the experimental results of the study, it was detected that the hybridization of glass fiber as the skin protection displays improved properties compared to the other composites followed by the hemp-epoxy composite. The mixture of the jute-hemp-epoxy composite is found to be acting better than the pure jute-epoxy composite. Test results showed that the hybridization reinforcement of jute with the hemp and jute-hemp with the glass improved the properties and can be considered as a possible replacement to glass fiber reinforced polymer composite in the minor applications. Uzay et al. [24] studied the capacity of impact energy absorption for several FRP (fiber reinforced polymer) composites. In the study, the experimental studies were made with the manufacture of specimens followed by subjecting them to Charpy impact test. The laminated composite plates were made-up with a hand lay-up vacuum bagging technique by using woven glass fabric layers, chopped glass fibers, and woven carbon fiber layers respectively. Blends of different fiber layers were also produced so as to see the improvement of the hybrid effect on impact energy. The test specimens were also subjected to a post-curing treatment process to examine the effect of post-curing temperature under impact loading. The absorbed energy of each FRP composite was determined by Charpy impact test consistent with ISO 179-1:2000 where impact strength values were calculated. Furthermore, the study introduced a relative comparison of specimens’ energy absorption abilities to find out a better combination of the laminated composite structure. Özturk, S. [25] has studied the mechanical properties like tensile, impact strength, hardness, and flexural stress of kenaf/PF (phenol-formaldehyde), fiber flax, and kenaf/flax hybrid PF composites as a function of fiber content. The tensile, impact strength, and flexural stress values of flax/PF composites were found to have low mechanical characteristics compared to kenaf/PF composites. The results of research show a decrease in flexural stress and impact strength for higher flax fiber composite. The above mechanical test results of kenaf/flax and flax PF hybrid composites
show that the hardness values have increased while the other quantities have decreased linearly with the increase of fiber content.

Wheat is considered a popular crop in agriculture industry. Its products like flour, starch are two main ingredients in food industry. However, some by-products like wheat straw are mostly used as food in animal growing sector. There are few research efforts that deals with wheat products. For instance, Reddy et al. [26] wrote a research paper focusing on preparing and investigating a polypropylene-based hybrid bio-composite and wheat straw and clay as reinforcement. The samples were prepared using melt bending technique. Throughout the study, hybrid composite components such as wheat straw, clay, and maleic anhydride grafted polypropylene as coupling agent varied to analyse their effect on flexural characteristics and water absorption. The study results demonstrate that the increase in the content of wheat fiber and clay increases the flexural modulus and decreases the resistance of water absorption. The increase in the content of coupling agent also increases the flexural modulus and the water absorption resistance.

This study aims to prepare a new bio-composite material by using sustainable raw materials then to investigate the effect of fiber mass content on the mechanical and physical properties of the polymer matrix composite obtained and conclude an optimum recipe for the production of such new composite materials. Some possible uses of such new composite material could be raw material, like boards, used in light furniture industry.

2. Methodology

2.1 Experimental procedure
In this work, a composite material consisting of wheat starch as a matrix and wheat fibers were prepared in the laboratories of College of Science, University of Kirkuk - Iraq. Wheat starch (available in the local markets) was used as a matrix material. While wheat fibers collected from rural areas around the city of Kirkuk, were used as reinforcement material. Glycerol was used as a compatibility agent, while sodium hydroxide (NaOH) was utilized as chemical treatment of fibers to provide a better adhesion between matrix material and fibers.

2.2 Preparation of fibers
Primarily, raw wheat fibers were chopped into short fibers with average length of 5.71 mm and average width of 0.38 mm using an industrial grinder. Chopped fibers were chemically treated by 0.5% wt. NaOH solution and then washed six times using tap water till reaching a neutral PH value of 7. Fibers were left to dry out for 24 hours at room temperature. Fibers then heated in an oven to a temperature of 160°C for six hours in order to completely remove the remaining moisture. Wheat starch was mixed with distilled water and glycerol with ratio of 1:1:0.6 by weight respectively. The compound is thoroughly mixed for 5 minutes until getting a homogeneous mixture.

2.3 Compounding and test samples preparation
Plasticized wheat starch is compounded with chopped wheat fibers at various fiber contents. For each 50 gm matrix material, the following masses of fibers were added 50, 60, 70, 80, and 90 gm, resulting a composite material with following fiber weight percent ratios of 50, 54.5, 58.3, 61.5, and 64.2 and a volume fraction of fibers of 81.82%, 84.35%, 86.29%, 87.79% and 88.97% respectively. A steel mold with length of 200 mm, width 55 mm and height of 100 mm was fabricated to contain the compound when exposed to molding pressure. The compounds first were cold pressed where compound is exposed to normal pressure of 100 bars for 30 minutes at room temperature. The second stage is heating the compressed specimen with the mold in a convection oven (Electro-mag, model: M 420) at 160°C for four hours. The resulting composite material is cut to the required dimensions for testing. All dimensions of specimens are chosen according to ASTM specifications for every test. The number of every ASTM standard is mentioned with the test performed.
3. Mechanical and Physical characterization

3.1 Mechanical tests
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Mechanical tests, under static or dynamic loading, reveal the characteristics of a certain material and its mechanical response under external loading. These tests are designed to guarantee that materials have appropriate properties and suitable for their planned applications. The tests conducted in this research are: Impact resistance, 3-point bending, and hardness test. All hot-pressed specimens are tested at room temperature.

3.1.1 Impact test
Charpy impact testing is considered one of the most widely used methods to simplicity and efficiently measure the relative strength of a material. This test shows the energy that a standard notched specimen absorbs while breaking by an impact force [27]. Impact test for specimens is performed to evaluate the improvement in fracture energy achieved using various fiber contents in the polymer. These tests are performed at room temperature with specimen dimensions in accordance with ASTM D256 [28]. A standard impact machine (Brooks LTD, model: MAT21) with a hammer mass of 30kg is used to conduct the tests. To confirm the accuracy of the test results, five identical samples are prepared and tested for each composite material. The average of the impact resistance value is reported in the results section.

3.1.2 Three-point bending test
The three-point bending flexural test provides a relationship between flexural stress and strain. The test is performed using different fiber contents of the composite material samples according to ASTM D7264 standards [29]. Tests of all five types of specimens are conducted using a standard three-point bending machine (Gunt, model: WP300). The formula used to evaluate flexural stress is given below [30]:

\[ \sigma_f = \frac{3FL}{2bd^2} \]  

Where,
F: bending load (N)
L: sample span length (m).
B: sample width (m)
d: sample thickness (m).

Flexural strain can be calculated using the following relationship [30]:

\[ \text{Flexural strain} = \frac{6Sd}{L^2} \]  

Where,
S: deflection measured (m)

3.1.3 Shore D hardness test
Hardness can be defined as a mechanical property of a material that refers to the materials resistance for penetrating forces. The main purpose of the hardness test is to provide information about the resistance of different versions of a certain material to penetration. Hardness test is performed on specimens of the composite materials according to ASTM D2240 standard [31] where hardness value is recorded after three seconds following hardness tester contact the specimen. These tests are performed using a calibrated Shore D durometer. Five different points on each specimen are tested then the average of hardness value is reported.
3.2 Physical properties

3.2.1 Density of the composite material
It is anticipated that the density of the composite material changes with the variation of the composition of the composite material. To quantify the change in density, both mass and volume measurements for five different specimens for every mixing ratio is taken, then the average density is reported. A precise electronic balance with accuracy of 0.01 gm (ST, model: 500) is used for mass measurements, whereas the dimensions of specimens is measured with a Vernier with accuracy of 0.01mm. The mass-density for each specimen calculated from following formula [32]:

\[ \rho = \frac{m}{V} \]  

Where,
\( \rho \) = mass density of the specimen in \( \text{kg.m}^{-3} \).
\( m \) = measured mass of the specimen (kg).
\( V \) = measured volume of the specimen (m\(^3\)).

3.2.2 Water absorption
Water absorption test is useful for evaluating the mass of water absorbed by a certain material under certain conditions. This test is performed according to (ASTM D570) standard [33] by recording the mass of dry specimen using a precise electronic balance. Then all specimens are immersed in distilled water for 13 hours. Mass measurements are recorded for every one hour until mass readings reach steady state values. Water absorption percentage is calculated using the following equation [34]:

\[ \text{Water absorption \%} = \frac{m_2-m_1}{m_1} \times 100 \]  

Where
\( m_1 \) and \( m_2 \) : masses of specimens before and after immersion in distilled water in (g).

3.2.3 Fibers length and width distribution
Optical image visualization and analysis for the wheat fibers before sample preparation is performed to evaluate the average length and width of chopped fibers that is used as reinforcement within the composite material. High resolution images are captured for the chopped raw material using high definition camera (Nikon, model: D5200). An image processing software (Klonk image measurement software, Image Measurement Corporation) is used to calculate the average length and width of fibers. Multiple measurements of known areas using this software showed an uncertainty of about 0.63%.

4. Results and discussions

4.1. Mechanical properties
The results of Charpy impact test are shown in Figure 1. The figure shows that the impact energy spent to break the composite material specimen decreases with increase in fiber weight content. This behavior could be mostly attributed to the fact that the matrix material, which is a type of natural polymers, has high capability to absorb shocks. Therefore, at low fiber content samples, which is also a high matrix content specimen, a higher impact values are seen. As fiber content increase, the brittle behaviour of fibers weaken the overall sample impact resistance. Results of three-point bending tests are shown in Figure 2. The figure depicts that the flexural stress increases as flexural strain increase at the beginning of all curves then all curves for all specimen flatten as strain increase. Also, there is a distinct variation among flexural stress-strain curves for different specimen types. In details, the higher the fiber content, the higher flexural stress the material can withstand. In addition, all specimens don't show a yield point which leads to the conclusion that the composite material is of a brittle nature and fail occurs without showing an elastic behaviour. Yield stress values extracted from Figure 2 by intersecting the tangent of every sample flexural stress and flexural strain curve at 0.002 flexural strain is shown in table 1. The data show that as the fiber
content increase the yield stress value also increase. This is an evidence that the current composite material can withhold higher stresses as fiber content increase.

![Figure 1. Influence of fiber content on average Charpy impact energy](image1.png)

![Figure 2. Flexural stress verses flexural strain for different specimen types](image2.png)

**Table 1.** Values of Yield stress calculated from flexural stress- flexural strain diagram.

| Fiber content (wt. %) | Yield stress (MPa) |
|-----------------------|--------------------|
| 50                    | 90                 |
| 54.5                  | 160                |
| 58.3                  | 190                |
| 61.5                  | 213                |
| 64.2                  | 235                |
Shore D hardness test results are shown in Figure 3. The figure illustrates that the value of the hardness is increasing from a value of 45.2 for 50 %wt fiber content to 68.7 for fiber content of 64.2 %wt. This behavior may be explained as fibers are harder material than base matrix material. Consequently, increasing fiber content will lead to thinner matrix material, as a result, the composite material gets higher penetration resistance then higher hardness value.

![Figure 3. Effect of fiber content on Shore D hardness](image)

4.2. Physical properties

Figure 4 shows density values calculated for composite material with different fiber content. The figure shows that there is an increase in density value by increasing fiber content in the composite. The highest density value recorded for 64.2 %wt. specimen is 1080 kg.m$^{-3}$ and smallest value recorded for 50 %wt. specimen is 1010.03 kg.m$^{-3}$). Increase in composite material density with fiber content can be explained as the fiber material is heavier and denser than the matrix material. Therefore, any increase in fiber content increases the overall density of composite material.

![Figure 4. Effect of fiber content on density of composite material](image)

Water absorption test results are shown in Figure 5. The figure shows that water absorption percentage increases rapidly for all specimen till reaching steady state values at about 8 hours from starting the test. For steady state water absorption values, the figure shows that the least water absorbent sample is the high fiber content sample, 61.5 %wt, whereas, the highest water absorbent sample is a lower fiber content sample 54.5 %wt. This behaviour could be explained depending on the fact that water absorptivity is affected by many factors like affinity of matrix material to water due to the existence of
gaps, holes, interfacial adhesion. At low fiber content samples, it seems that matrix material can contain more moisture than higher fiber content samples where fiber existence seems to reduce the matrix tendency to absorb water where the result is lower water absorption. Changes in the dimensions of specimens were negligible except for the 64.2 %wt specimen in which there was a slight increase in thickness of about 7%.

![Figure 5](image_url)

**Figure 5.** Effect of fiber content in weight percent on water absorption

Figures 6 and 7 show data for both chopped fiber length and width distribution respectively. The figures show a clear variation of length within the values of 1.4mm- 14mm. The average length calculated for 100 fibers is found to be 5.71mm with standard deviation of 3.64mm. On the other hand, Figure 7 shows wider width distribution between values of 0.16mm- 0.86mm. The average width calculated for 100 fibers is 0.38mm with standard deviation of 0.01mm.

![Figure 6](image_url)

**Figure 6.** Chopped fiber length distribution for 100 raw fiber sample
Figure 8 shows a microscopic image for the 50gm composite material specimen at 1000x magnification. The photo shows clearly different fiber layouts in combination with matrix material. In addition, the variation of fiber length and width is obviously noticed. Moreover, the photo shows a clear homogeneity and adhesion between fiber reinforcement and matrix material which is a result of high compression used in the process of material preparation.

Figure 7. Chopped fiber width distribution for 100 raw fiber sample.

Figure 8. Microscopic image at 1000x magnification for the 50 %wt composite material sample.

5. Conclusions
In this study, the mechanical and physical properties of a bio composite material prepared from wheat fibers as a reinforcement and wheat starch as a matrix is studied. Conclusions from this study can be summarized as following: The Charpy impact energy values decreases with an increase in fiber content. Three-point bending test results show that as higher fiber content as higher flexural stress the material can withstand. An increase in hardness values is seen as fiber content increase. Also, the density of composite material increases as fiber content increase. Whereas water absorption decreases by
increase in fiber content. The overall conclusion from this study is that the combination of wheat starch-wheat straw fibers can provide a composite material with mild mechanical and physical properties. Also, best combination of composite material ingredients is 64.2 %wt fiber content which gives the highest mechanical properties except for Charpy impact test where strongest composite material sample was the 50 %wt fiber content.

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