Experimental Investigation of Natural Fiber Reinforced Polymers

BY

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Acknowledgment

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Abstract

There is a growing use of polymers in different applications worldwide. This phenomenon resulted in an enormous increase in the amount of plastic waste. These wastes have significant impacts on the environment including pollution and depletion of resources. Recycling plastic waste and reusing it helps in alleviating environmental degradation. The amount of recycled plastics all over the world not utilized is huge. U.S.A recycled 5.7% of the total plastics generated and Western Europe recycled 39% of total amount of plastics consumed [1].

This research studies the potential usage of virgin and recycled polymer reinforced with natural fiber such as rice straw to produce high value products that have technical, economical and environmental demand. The use of fiber reinforced polymer became popular during the last ten years. The reinforcing material is embedded in the matrix material to improve its mechanical and physical properties; reinforcing plastic waste with natural fiber is a new trend to enhance the mechanical properties of this composite material. In this study, a composite material was developed using Low density polyethylene (virgin and recycled) as a matrix reinforced with treated rice straw. Tensile and flexural behaviors of the synthesized composite were investigated. The results were promising showing an enhancement in the mechanical properties of the virgin polymer composite compared to the virgin polymer. The flexural stress of the composite increased three times the virgin flexural stress. While the tensile stress increased from eight times the original tensile stress. However the enhancement for the recycled composite represented two times the original flexural stress and four times the original tensile stress.

In an attempt to improve the mechanical properties of the recycled polymer composite, rice straw treatment through carbonization as a reinforcement was investigated and proved higher tensile stress than the chemically treated rice straw by eight times the tensile stress.
recycled treated and non carbonized fiber, thus approaching the value of the virgin LDPE composite.

With this new material produced entirely out of waste, this product will reduce the detrimental problem of solid waste to the environment including saving energy, water and cost. The problem of depletion of natural resources will be minimized. The current practice of disposing plastic waste will decrease as waste will be utilized with high quality. Also the current practice of burning rice straw will be restrained. Finally, an innovative, clean, cheap and effective yet simple technology with different procedures was introduced in this thesis research to determine the suitability of fiber reinforced composites techniques worldwide.
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**Nomenclature**

CDM: Clean development mechanism

DOE: Design of experiments

FRP: Fiber reinforced polymer

H$_2$O$_2$: Hydrogen peroxide

H$_2$SO$_4$: Sulphuric acid

H$_3$PO$_4$: Phosphoric acid

LDPE: Low density polyethylene

MSW: Municipal solid waste

NaOH: Sodium hydroxide

NaOCl: Sodium chlorite

PVC: Polyvinylchloride

RLDPE: Recycled low density polyethylene

SEM: Scanning electron microscope

VLDPE: Virgin low density polyethylene
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1.1 Sustainable Development

Sustainability is to use the natural resource on hand in a way that keeps the environment clean and safe in addition to maintaining these resources for the next generations. Since the 1980s, sustainable development had become very important for human beings and of great concern. It is the development that meets the needs of the present without compromising the ability of future generations to meet their own needs [2]. The concept of sustainable development required the reconciliation of the three demands that are known as the three pillars and these are environmental, social and economic demands as shown in figure (1).
The main source on which human beings depended was the agricultural life. The effect of lighting fires and looking for special types of food slightly altered the environment composition of animal and plant communities. The real problem was when the industrial revolution started in Europe, which meant that a substitute for fossil fuels had to be found to power sophisticated machinery. By the 20th century the industrial revolution had lead to an exponential increase in human consumption of resources as well as an increase in health, wealth and population [2]. The problem of sustainable development of green issues goes back to the 50s and the 60s. These eras were not valuable to the development of the environmental education; moreover, it was not until the 70s when these issues began to emerge on the governmental scene with the 1972 Limits to Growth Report and the Stockholm Conference. During this period of time, strong public awareness of the environment as this was the decade during which Greenpeace was founded. In 1970s the main concern was about energy supplies, during that period there had been a number of international efforts to control the industries and their achievements regarding the environmental and sustainable ideals. In 1980s the gap between what had been expected by the public from the industries and what the industries were actually doing in terms of the environment were the results of Global warming. Moreover, there has been a loss of habitats for wild animals and a decrease in natural resource due to exploitation of forests [3]. The main reason behind people’s concern about sustainable development is that the materials on earth are finite. Sustainable development aims to establish a system of resource consumption that both meets the needs of human life and leaves the environment healthy enough to continue to produce the resources future generations will need.

In order to tackle the problem of sustainability, a methodology that is efficient, useful, economical, effective, and easy to use should be implemented. William McDonough and Michael Braungart developed a methodology titled "Cradle to Cradle" that promotes the
concept of zero pollution [4]. This opposes the concept of "Cradle to Grave" which is commonly used whereby the waste is burned, incinerated, or landfilled. This essentially causes air pollution with numerous toxins being dissipated into the air as a result of waste burning; water pollution with chemicals from improper landfills or garbage sites disseminating into rivers, lakes, or even groundwater; and land pollution with the occupation of land areas for landfiling that could be better utilized for either urbanization and housing or for agricultural purposes. The concept of Cradle to Cradle is done partially by utilizing the waste of one process as the raw material for another process. Thus the phrase "Waste = Food" is investigated to promote the utilization of all forms of waste as useful materials for another process. This does not simply mean recycling. On the contrary, the 'Cradle to Cradle' concept wishes to utilize the waste of a process directly as the raw material for another, undergoing a circle of resources that are ultimately never wasted and thus never depleted. Such examples could be as simple as using organic waste, such as orange peels for instance, as feed for animals on farms. Another example could be the utilization of animal waste and excrement for compost fields to be later used as fertilizer for agricultural products. By these simple examples waste is eliminated and utilized it to enhance the mechanism of another process. Not only is this mechanism efficient and effective, but extremely economical. The disposal of waste is actually quite costly, yet with this mechanism the raw material for a process is not bought at an expensive rate but rather taken from the waste of process that is no longer needed [5].
The concept of cradle to cradle is displayed in figure (2) using the "industrial ecology" methodology where the waste of one product, is the new raw material utilized by another product. For example if the diagram is followed from the hotels and housing, it can be seen that the garbage produced there is collected and sorted. Each material undergoes its own line or path to become something new. Food waste goes to the animal farms to feed them. The animals produce excrement which then goes to function as compost. The diagram can be traced for all materials reaching a closed loop of resources where there is an effort to approach zero waste in this tourist resort in the future. The differences between "Cradle to Cradle" and "Cradle to Grave" are shown in Table (1)
Table 1: Comparison between "cradle to cradle" Vs. "cradle to grave" [5]

<table>
<thead>
<tr>
<th>Cradle to Cradle</th>
<th>Cradle to Grave</th>
</tr>
</thead>
<tbody>
<tr>
<td>Zero Pollution</td>
<td>Air, Water, Land Pollution</td>
</tr>
<tr>
<td>No cost of Disposal</td>
<td>Expensive cost of disposal</td>
</tr>
<tr>
<td>Low cost of raw materials</td>
<td>Expensive cost of new raw materials</td>
</tr>
<tr>
<td>Minimize health risks</td>
<td>Colossal health risks</td>
</tr>
<tr>
<td>Efficient use of land</td>
<td>Land is wasted for landfill</td>
</tr>
</tbody>
</table>

The benefits of utilizing the “Cradle to Cradle” are eliminating health risks water and air pollution. It helps in minimizing Ozone depletion, global warming and landscape degradation within efficient utilization of resources. Moreover this methodology reduces solid and liquid wastes thus preventing resource depletion and acidification of natural and built environment. This helps in minimizing the cost of waste disposal, cost of raw material and cost of health bills. According to McDonough and Braungart, this methodology can be applied to any industry for any material. The material studied in their book is a synthetic paper that does not use any wood pulp or cotton fiber but is in fact made from plastic resins, inorganic fillers and marble waste. It is waterproof, durable, and thus becomes a "technical nutrient" as a prototype that can ultimately achieve zero pollution and save our planet from deterioration and degradation economically and swiftly [4].

An example of sustainability can be observed in the recycling process for waste products. This is done through a recycled polymer which produces products with properties that are quite low compared to virgin polymer. The way to improve the recycled polymer is to add natural fibers acting as reinforcement. While implementing sustainable development, it is
very important to be able to quantitatively measure sustainability as it becomes easier then to set goals, apply management strategies, and measure progress.

1.2 Composites

Research interest has been shifting from monolithic materials to fiber reinforced polymeric materials. Composite material is made of the combination of two different materials to achieve certain properties different from each material on its own. One of the two materials acts as a matrix, while the other acts as reinforcing material. The reinforcing material is imbedded in the matrix material to improve its mechanical and physical properties. The ancient composites are bricks which are made of mud and straw. One of the most common composites is asphalt concrete. Figure (3) shows the types of composites [6]. The focus of this thesis is fiber reinforced composite using short and randomly oriented natural fibers.

Mechanical properties of composites vary according to the matrix and reinforced materials used. The main focus of this thesis will be the fiber reinforced polymers (FRP) due to their superior properties: low maintenance requirements, high stress to weight ratio, high corrosion, impact resistance, non conductive, avoid electrical hazards, reduced cost, easy
installation due to light weight and Fire retardant. FRP materials are used in many industries such as aerospace components (tails, wings, propellers), bicycle frames, boat hulls, fishing rods, storage tanks, baseball bats, ice skating boards, door panels, automobile industry, construction material for buildings, marine application and sporting goods industry [7].

1.3 Fibers

It is a class of materials that are continuous filaments or discrete elongated pieces, similar to lengths of thread. They play an important role in holding the tissues of both plants and animals. Human uses fibers into filaments, string or rope, a component of composite materials, or matted into sheets to make products such as paper or felt. Synthetic fibers (glass, carbon, aramid) can be produced in large amounts. The problem with synthetic fibers is the environmental factor as well as the cost while natural fibers (cellulose, rice straw, palm, kenaf) are cheap and environment friendly [8].

1.3.1 Natural fibers

The reinforcing material used in this research is natural fibers. They are composites of hollow cellulose fibrils held together by a lignin and hemicelluloses as shown in figure (4). Each fibril consists of a thin primary wall around a thick secondary wall. The secondary wall is made up of three layers and a thick middle layer. This layer determines the mechanical properties of the fiber. The middle layer consists of a series of helically wound cellular micro fibrils made of long chain cellulose molecules [8]. The following table (2) shows the properties of several natural fibers including the rice straw that is the focus of this research. The properties of rice straw are not the highest rank but it is chosen to help minimizing the pollution produced by burning rice straw which will be discussed in greater detail later.
Table 2: Mechanical properties for natural fibers for composite applications [8]

<table>
<thead>
<tr>
<th>Fiber</th>
<th>Tensile stress (MPa)</th>
<th>Elongation at break (%)</th>
<th>Young modulus (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flax</td>
<td>300–1500</td>
<td>1.3–10</td>
<td>24–80</td>
</tr>
<tr>
<td>Jute</td>
<td>200–800</td>
<td>1.16–8</td>
<td>10–55</td>
</tr>
<tr>
<td>Sisal</td>
<td>80–840</td>
<td>2–25</td>
<td>9–38</td>
</tr>
<tr>
<td>Coir</td>
<td>106–175</td>
<td>14.21–49</td>
<td>4–6</td>
</tr>
<tr>
<td>Oil palm</td>
<td>130–248</td>
<td>9.7–14</td>
<td>3.58</td>
</tr>
<tr>
<td>Hemp</td>
<td>310–900</td>
<td>1.6–6</td>
<td>30–70</td>
</tr>
<tr>
<td>Wool</td>
<td>120–174</td>
<td>25–35</td>
<td>2.3–3.4</td>
</tr>
<tr>
<td>Cotton</td>
<td>264–800</td>
<td>3–8</td>
<td>5–12.6</td>
</tr>
<tr>
<td>Rice straw</td>
<td>69–100</td>
<td>9.4–12</td>
<td>27–30</td>
</tr>
</tbody>
</table>
Natural fibers are divided into botanical fibers, chemical and commercial fibers. The botanical fibers are important constituents of wood. The fiber cells have very thick walls and they occur in bundles thus closely cemented together. The chemical fibers consist primarily of nearly pure cellulose, hemi cellulose as flax, hemp; ramie the cellulose is associated with pectic material, while Jute fibers are more lignified. High percentage of cellulose in fibers correlates positively with stress and durability. The commercial fibers are small, thin, slender fragments of many substances. They can be divided into mineral origin, asbestos, spun glass, animal origin, wool, silk, hair, feathers and plant origin. The main function of the fiber is to carry load, provide stiffness, stress, thermal stability and provide electrical conductivity. Natural fibers are a healthy choice as they provide natural ventilation and act as insulators against both cold and heat. Coconut fibers have natural resistance to fungus and mites and hemp fiber has antibacterial properties, and studies show that linen is the most hygienic textile for hospital bed sheets. They are also a responsible choice as they are of major economic importance to many developing countries and also they have a safe handling and non abrasive. They are a sustainable choice as they are a renewable resource. They are carbon neutral which decrease pollution by decreasing release of carbon. Moreover processing produces residues that can be used in bio composites for building houses or to generate electricity. They are a high tech as they have good mechanical stress by drawing the fibers, low weight, low density and low cost [8].

In Europe, the automotive industry is using an estimated 80,000 tons of natural fibers a year to reinforce thermoplastic panels. They are of low cost, low energy consumption, non abrasive nature, safety in handling, low density, potentially higher volume fraction, superior specific properties. Bio fibers such as hemp, jute, kenaf, sisal, and bamboo can potentially replace glass fiber to enforce polymeric resins. Disadvantages of natural fibers
include low resistance to moisture, seasonal quality variations. These issues are being addressed in order to achieve commercially viable bio fiber filled composites for automotive and other demanding engineering applications [8].

**1.3.2 Rice straw**

A natural fiber that is environmentally controversial nowadays is rice straw. Rice straw posed numerous environmental problems all over the world after the paddy fields are harvested. The main reason is because of the burning of rice straw. Rice straw is the by-product of rice once the grain and chaff have been removed. In Egypt, the problem of effectively clearing up rice straw is significant. Most of the rice straw end by being burnt through ‘open burning’ by the farmers. This has resulted in several negative implications including the ‘black snow” phenomenon that had caused major road accidents. New and innovative usage of rice straws will greatly help in overcoming the environmental issues as an effective discharge of these waste materials rice straw which is about 40 percent, and the burning of 500,000 tons of rice straw may return 200,000 tons of carbon into the atmosphere. This carbon is fixed during the growing season by photosynthesis and there is little net gain. If the straw is incorporated in the soil it increases methane emissions. This is more damaging than the byproducts of burning. Methane is a special concern for global warming, because each methane molecule has 20 times the heat capturing potential of a carbon dioxide molecule. Even allowing for the lower level of emissions, the net impact on global warming would be 10 times worse than the effects of carbon dioxide from field burning [9]. Moreover smoke can cause health and safety problems, including asthma, allergies, bronchitis, and respiratory distress. Smoke can also contribute to highway accidents. In developed countries, rice straw burning in the fall is spread over a period of weeks or months and is regulated to discourage burning when meteorological
conditions are likely to lead to smoke accumulation. Health risks are minor due to burning management, but may be significant locally near burns. These risks include exposure to the various gases and particles created by recombination of gases, ash and dust raised from the soil surface. The obvious problems associated with rice smoke led to restrictions on burning beginning in 1971 under provisions of the Health and Safety Code, these were revised and made more flexible in the early 1980s[9].

Rice straw has several applications including usage as reinforcement in polymers, composting and animal fodder. 1-5% sodium hydroxide increased the protein content to more than 8%. Ensiling rice straw with sodium hydroxide decreased the crude fiber content relative to that in the untreated rice straw. The feed intake of rice straw when fed alone was very poor but it increased significantly with the addition of 5% molasses. Addition of urea and molasses enhanced feed intake more than the addition of molasses [10]. A special treatment with an acid or an alkali is needed when rice straw is used as reinforcement in order to improve the properties of rice straw. This treatment is considered a physiochemical treatment as a chemical is added to improve the physical properties of the fiber. This treatment is done with 1% or 5% concentration of phosphoric acid. The acid has to be chosen carefully. The use of hydrochloric acid deteriorates the mechanical properties of the fiber including poor interfacial interaction. This leads to internal strains, porosity, environmental degradation, moisture absorption, debonding over time. This percentage is chosen according to several researches mentioned in the literature review. The fiber treatment produces surface modification, improved thermal stability of fiber, improved fiber–matrix adhesion allowing an efficient stress transfer from matrix to the fibers. Moreover the tensile stress of composites with treated fibers is raised to around 50%. This is due to the shrinkage process during treatment of fiber that has an effect on the fiber structure [11].
1.4 Matrix

The main function of the matrix is to bind the fibers and transfer the load to the fibers. The idea of using polymers with fibers was highly recommended due to the superior plastic properties including lighter weight, extreme durability, resistance to chemicals, water and impact, excellent thermal and electrical insulation properties. The issue of producing FRP composites using virgin or recycled plastic is controversial. Various opinions were found regarding the practicality of usage, mechanical, physical properties and final appearance. However, studies based on recycled products are limited due to the fear of obtaining a product with non controllable physical and mechanical properties resulting from degradability and impurities leading to decrease the mechanical behavior of the composite.
Chapter 2

Literature Review

2.1 Plastic Industry

The considerable growth in plastic use as shown in figure (5) is due to the beneficial properties of plastics mentioned previously. The plastic industry existed about fifty years ago. They are now available in all our daily life products: pillows, mattresses, cars, computers, wires and electric cords. The building and construction industry uses vinyl siding for homes because of its appearance, durability, ease of installation and energy efficiency. Even though plastics have a variety of benefits, they are in fact detrimental to the environment. Many of the environmental impacts associated with the production and manufacturing of plastics include the fact that plastic production consumes a large amount of energy and materials, primarily fossil fuel that when combusted emits toxins into the air that are hazardous to the health of civilians. It is estimated that 4% of the world's annual oil production is used as a feedstock for plastics production and an additional 3.4% during manufacture. Plastics production also involves the use of potentially harmful chemicals, which are added as stabilizers or colorants. Many of these have not undergone environmental risk assessment and their impact on human health and the environment is currently uncertain. An example of this is phthalates, which are used in the manufacture of Polyvinylchloride (PVC). PVC has in the past been used in toys for young children and there has been concern that phthalates may be released when these toys come into contact with saliva, if the toy is placed in the child's mouth or chewed by the child. Risk assessments of the effects on the environment are currently being carried out. Other environmental impacts of plastics include the extensive amount of water that is needed in
manufacturing. Also, the numerous plastic bags that are dispersed as litter in urban areas have also become a plaguing concern. Due to the magnitude of the problem associated with plastic, the world is now directed towards the use of recycled plastics. It has been shown that there are numerous benefits to the recycling of plastics: reduce water usage by 90%, reduce CO₂ emissions by two and half times and reduce energy consumption by two third [5].

Figure 5: Growth in the plastic industry worldwide [12]

2.2 Plastic recycling

The market potential regarding the usage of plastic waste into other utilizations is huge due to the high amounts of its disposition which constitute the largest share of the global municipal solid waste (MSW). In the United States, the waste of plastic; in 2005, was calculated as 11.8 % of 246 million tons of MSW generated. In India, plastic in MSW makes up to 9-12 % by weight of the total in addition to other wastes that may contain higher proportions of plastics. The majority of plastic waste generated is disposed. However the continuous growth of worldwide plastic consumption due to its short life
cycle compared to other products; roughly 40% have duration of life cycle smaller than 1 month, and the legalization of many countries concerned with minimizing landfills content and incinerators led to a necessity of recovering plastic waste instead of disposing [13]. Incineration and land filling alternatives were rejected by several countries due to their potential danger to the environment either by polluting air or land; which result in not closing the cradle to cradle loop and depleting natural resources [6]. Plastics account for an increasing fraction of municipal solid waste around the world. In the United States, plastic had a total volume of 19.2 million tons in 2001, accounting for about 8.4% of total municipal solid wastes. Thus, used plastics are becoming a potential worldwide source of raw materials. As a consequence the tendency towards recycling has increased resulting in attempts for plastic recovery. In 2004, 8.25 million tons (39% of total amount of plastics consumed) in Western Europe; 35,000 tons (13.48 of total imported virgin plastics) in New Zealand were recovered. While in 2005, the United States recycled around 5.7% of the total plastics generated. On the other hand, some states in the US like Michigan have a recycling rate close to 100%. In Brazil, potential in recycling have been raised where 15% of all plastics consumed are recycled and returned to industry [1, 14].

Even though the technologies and advancements with respect to recycling plastics have soared, there remains the unanswered question of cost. However according to a study entitled "The Cost of Reducing Municipal Solid Waste" done by Resources for the Future foundation, it was shown that plastic recycling pays back once it is done in large quantities. If it is to be done for a whole community or district the pay backs may be more obvious. Yet when the quantities are much lower, the cost becomes an obstacle due to energy cost, transportation cost, sorting, labor cost. The importance of recycling plastics cannot be overstated as its benefits are plentiful [15]. It could be reinforced with natural fibers such as rice straw. Rice straw can work well with both VHDPE and RHDPE as
reinforcing filler. This might imply the application prospect of conveniently introducing rice straw into thermoplastic composite industry. There is no reason to believe that the use of recycled HDPE as a matrix has had deleterious effects upon the tensile stress and flexural stress as there is a slight decrease in tensile and flexural stress between virgin and recycled composites [12].

2.3 Applications of natural fiber reinforced polymers

A polymer is generally manufactured by poly condensation, polymerization or poly addition. When combined with various agents to enhance or in any way alter the material properties of polymers the result is referred to as a plastic. Composite plastics refer to those types of plastics that result from bonding two or more homogeneous materials with different material properties to derive a final product with certain desired material and mechanical properties. Fiber reinforced plastics are a category of composite plastics that specifically use fibrous materials to mechanically enhance the stress and elasticity of plastics.

There are several applications for fiber reinforced plastics that are very useful these days in industry. The use of rice straw as reinforcement in polypropylene composites with 20 and 30% by weight was successfully prepared by extrusion and compression molding. The results of the mechanical properties showed that rice straw can be used as an alternative reinforcement for polypropylene. Higher tensile modulus was obtained for composites containing higher rice straw content. The renewability of rice straw and the recyclability of thermoplastic polypropylene provide an attractive eco-friendly quality to the resulting composites [16].

Underground pipes, water supply pipes rags pipes, shield pipes of communication cables as shown in figure (6). The rehabilitation of those damaged underground pipes is
imminent. The material used for the new pipes is natural fibers reinforced composite, the natural fiber used is jute or rice straw. These natural fibers are cheap with high resistance to corrosion and thermal stability. They have the advantage of avoiding contamination of water in the pipes unlike glass fibers [17].

The highway infrastructure has been deteriorating for many years, a result of harsh environmental conditions, heavy loads, insufficient maintenance, In addition to high traffic volumes, tight construction budgets, and challenging roadway construction areas that have put a strain on the ability of conventional materials to meet the public needs for rapid construction, long lasting structural components, and lightweight, easily constructed facilities. Fiber reinforced polymer (FRP) composite materials, which have been used for some time in the aerospace and military communities, have been perceived as a potential solution to some of the highway community’s infrastructure needs because FRP’s stresses mesh with the shortcomings of several traditional materials. During the past decade, a significant amount of basic research has been conducted in the United States and abroad on the use of FRP composite materials for highway infrastructure applications [18].
Using polymers instead of aluminum is taken into account in the production of engine intake. They are now made from glass fiber reinforced Plastics as it is light in weight than aluminum and has improved surface quality and aerodynamics. Reduction in components by combining parts and forms into simpler molded shapes is easier in plastics than Aluminum. Pedals can be molded as single units combining both pedals and mechanical linkages simplifying the production and operation of the design [13].

FRP composites are attractive for use in civil engineering applications due to their high stress to weight and stiffness to weight ratios, corrosion resistance, light weight and potentially high durability Their application is of most importance in the renewal of constructed facilities infrastructure such as buildings, bridges, pipelines, etc. Recently, their use has increased in the rehabilitation of concrete structures, mainly due to their tailored performance characteristics, ease of application and low life cycle costs. The success of structural rehabilitation measures led to the development of new lightweight structural concepts utilizing all FRP systems or new FRP/concrete composite systems [19].

FRP doors are produced in different lengths they have an easy workability, durable, impact resistant, water repellent, chemical Resistance and stain proof. FRP roof rainwater gutters are produced to come along with support brackets in various lengths and are used in a vast array of industries. These gutters are fitted on the roof of industrial building for water drainage. FRP gratings are used in chemical, beverage, aircraft, and food applications. They have non-skid surfaces which have non corrosive nature, non-magnetic, non-conducive, lighter than aluminum, 50% stronger than hot rolled steel, low thermal expansion as compared to aluminum and steel, low maintenance and easy installation as shown in figure (6) [20].
2.4 Chemical treatments of rice straw

The enhancement in mechanical properties including flexural and tensile stress in alkali treated fibers is attributed to improved wetting of alkali treated with matrix. The removal of impurities and waxy substances from fiber surface and creation of rougher topography after alkalization thus quality of fiber will be promoted also content of hemi cellulose lignin decrease so increase effectiveness of oriented cellulose fibers. Alkali improved dispersing of fiber in matrix which resulted in increase in fiber aspect ratio this increased fiber reinforcement effectiveness. The fibers that can be treated with alkali solutions are jute, palm, wood, paper and rice straw. A solution of 5% NaOH concentration was used for two hours at 150\(^{\circ}\)C washed with distilled water until NaOH was eliminated. Fibers are dried at 60\(^{\circ}\)C for 24 hours. The increase in the chemical concentration of the alkali damages the fiber structure. When soaking for longer time give better results due to improved crystallinity of fibers and remove of hemi cellulose lignin [21]. Another alkali treatment for sisal fibers is done by using between 4 and 10% NaOH solution for 1 hr at room temperature. The diameter, weight of fibers was found to decrease due to removal of lignin. Alkali increase the roughness of the fiber surface hence increasing the surface
area available for contract for matrix. The fiber matrix adhesion increased with this treatment [22].

Adding sodium hydroxide and starch ethylene vinyl alcohol to 10 grams of rice straw improved the thermal stability of fibers, compatibility and interfacial bond stress. Tensile stress improved by 53% due to lack of impurities. Treatment in the water also improved values of Young’s modulus by 75%. The treatment also improved thermal degradation of fibers. These changes can be interpreted in terms of removal of hydrolyzed substances which decompose earlier than cellulose lignin leading to higher thermal stability of second step of degradation. Crystallization of fiber increases due to rupture of linkage lignin and alkali [21]. Another treatment is adding 1% concentration of NaOH for 3 hrs at room temp to rice straw, then washed and over dried at 100°C for 72 hrs then cleaned. The phenomenon of split fibers breaks fiber bundles into smaller ones by dissolution of hemicelluloses. This increased the effective surface area available for contact with matrix so interfacial bond improved. Pull out effect decreased in treated fibers and reduced water uptake of composite system [23].

The Coconut fiber has the same properties of rice straw. Five grams of the fiber was chemically treated by three treating agents: Sodium hypochlorite [NaOCl], sodium hypochlorite/ sodium hydroxide [NaOCl/NaOH] or Hydrogen peroxide [H₂O₂]. The effect of these treatments on the structure, composition and properties of fibers was studied using SEM. It showed that treatment with H₂O₂ is the most efficient in terms of waxy and fatty acid residues removal but it does not modify the surface chemical composition, Moreover it revealed a reduction of the hemicelluloses content in the fibers treated with NaOCl/NaOH. Consequently, this fiber showed a greater exposure of cellulose and a reduction in thermal stability. This treatment was disregarded because the thermal stability is needed in several applications of FR [24].
Four concentrations of sulphuric acid [$H_2SO_4$] were used (1-4%) with 5 grams wheat straw at three different temperatures (98-121-134 °C). The predominant effect was to solubilize the hemicelluloses fraction of wheat straw. This was achieved by using the highest temperature. The solubility reached 67% but there was no significant improvement in the thermal stability and interfacial bond. But there was an improvement in tensile stress of FRP by 40-50 % with the concentration of 1 % $H_2SO_4$. This result promotes a cleaner production technique. Using 1 % instead of 4 % leads to reduction of chemicals at the source thus minimizing cost [25]. Another treatment of rice straw was done using a sulfuric acid concentration of 1.5 for microbial production of Xylitol [25].

There are some physical and chemical treatments of natural fibers including rice straw to avoid the deficient parameters. Some of the natural fibers have poor wet ability incompatibility with some polymeric matrix and high moisture absorption and poor adhesion. This means that the full capabilities of the composite cannot be exploded and it is vulnerable to environmental attacks. This weakens composite and reduce its life span there are two Physical treatments: cold plasma treatment, and corona treatment. The latter is a technique for surface oxidation activation. It changes the surface energy of the fiber. The chemical treatment can be done using maleic anhydride, organosilanes, isocyanates, sodium hydroxide, permanganate and peroxide. These coupling agents eliminate weak boundary layers, produce a tough, flexible layer, develop a highly cross linked inter phase region, improve the wetting between polymers and form covalent bonds with both materials. The tensile stress of the maleic anhydride treated fiber composites was found to be higher than the untreated fiber. The jute – epoxy composite when treated with Silane proved to have better interfacial bonding and hence better mechanical properties specially adhesion. An example of chemical treatment is adding wax. Wax enhances natural fiber reinforcement plastics by overcoming processing problems caused by cross linking and
decomposition. The wax has low velocity to enable impregnation. The mechanical properties of composites increased with these treatments but increasing the treatment cause an elevation of costs [26].

Mechanical testing of these chemically treated natural fibers includes tensile test and flexural test are the performed tests in this research. The standard specimens are tested at a rate of (2 -5mm /min). The flexural stress, failure strain and tensile stress can be calculated from stress strain curve. Sample standard dimensions according to ASTM are (40- 100 mm) length (10-20 mm) width and (2-5mm) thickness [27].

2.5 Fiber treatment through carbonization

Fiber treatment through carbonization is another treatment for rice straw fibers in which heating without air occurs. The pore structure and adsorption properties of carbonized fibers are strongly used in several applications due to improved mechanical properties. The steps of carbonization include thermal treatment of raw fibers in an inert atmosphere followed by an activation step with CO2 or steam at the same temperature used. In chemical activation, the raw fibers were impregnated in a solution of phosphoric acid and heated at 900 °C in an inert atmosphere. This chemical treatment leads to a high porosity, which enables a high adsorption capacity for micro pollutants. Moreover, it produces numeric acidic surface groups involved in the adsorption mechanism of dyes and metal ions. The phosphoric acid promotes the bond cleavage in the biopolymers and dehydration at low temperatures, followed by extensive cross linking that binds volatile matter into the carbon product and thus increase the carbon yield [28].
2.6 FRP Manufacturing Techniques

Various techniques were adopted in literature to manufacture FRP, however; the two main adopted techniques are extrusion and injection molding. Typically, the extrusion process produces continuous linear profiles via forcing a melted thermoplastic through a die; on the other hand, the injection molding process produces three-dimensional items with minimizing the stages of post-manufacturing [29].

The manufacturing techniques adopted by Bengtsson and Oksman, were based on feeding the plastic granules to the cororating twin-screw extruder at temperatures varying from 165 to 200 C. A rectangular die was used at the extruder end and the extrudates were then cooled at ambient temperature. Silane was added during extrusion to enhance the product properties. They showed that adding silane resulted in superior increase in toughness, impact strength and creep properties in comparison to those without silane; However, The flexural modulus was lower [30].

A comparison was conducted between extrusion and injection molding for producing WPC, common steps found in both include melting, shaping, and cooling; in addition, they both use screws to convey, pump, and blend the mixed component. However, they added that process parameters such as residence time, temperature, pressure, shear rate, shear stress, and cooling rate are different. Moreover, they concluded that pressure and shearing in injection molding are higher than en extrusion regardless the process parameters mentioned [29].
2.6 Objective

Plastic wastes are a major environmental concern that needs to be dealt with to minimize the amount of municipal solid waste, depletion of natural resources and enhancing the sustainability concept for future generations. As a consequence, the purpose of this study is to take advantage of these useful wastes. The objective of this thesis research is to enhance the properties of plastic waste through reinforcement with rice straw, using a simple and effective technology. Rice straw will be treated using acids, alkali and carbonization treatment. The mechanical properties of rice straw will be investigated including flexural, tensile stress and elastic modulus. The composites (virgin and recycled) will be compared with the polymer without reinforcement to observe the effect of reinforcement on the mechanical properties of the composite.
Chapter 3

Methodology

3.1 Goal of work

The current practice of disposing plastic waste and burning of rice straw increased the rate of pollution rapidly. Moreover the rate of depletion of earth natural resources is growing enormously. This led scientists to think of efficient ways for waste utilization. Recycling plastic waste and enhancing its properties through reinforcement with natural fibers is a successful way to achieve zero pollution. The goal of this work is to determine the best treatment approach of the fiber, length of fiber, concentration of fiber in the composite. Two different categories of plastic were used, VLDPE and RLDPE. Polyethylene was selected for testing procedures as it is soft, flexible and inert, thus avoid reacting with any other elements. It also has a low static charge so it does not attract dust and dirt. The reinforced fiber is treated chemically by phosphoric acid, sodium hydroxide and sulphuric acid to enhance the properties of fiber. Another treatment of fibers that is used is carbonization. These treatments not only enhance the mechanical properties of fibers but also the composites properties.

3.2 Experimental procedure

The manufacturing processes of FRP with either virgin or recycled polymer were performed at The American University in Cairo’s Environmental Management Labs. All the processes have gone through several pilot experimentations till reaching the procedures mentioned above in the experimental procedure section. The second step is to mix the
treated rice straw fiber with the polymer then using extrusion and compression molding to produce the samples as shown in figure (8).

Figure 8: FRP process flow chart

The process starts by cutting different lengths of rice straw fibers through a shredder. The fibers are treated with 1% phosphoric acid. The scale used to weigh the fibers was calibrated using linear regression method based on standard weights brought from the polymers lab at the American University in Cairo. The regression equation obtained for calibration is \( x = \frac{(y+0.034)}{0.999} \) where \( y \) is the dependent variable consisting of the measured weight in grams and \( x \) is the independent variable of the calibrated weight. The equation was applied to the weights measured and calibrated readings were obtained. The weights used were (100, 50, 20, 10, 5, 2)

Based on literature review, the fiber was soaked for 2 hours at 150 C. the treated fiber is dried in a furnace as mentioned in the experimental procedures section. It was assured that the moisture was totally eliminated. The fibers were weighed every hour. After 8 hours, the
weight did not change assuring that the fibers are dry. This procedure was repeated 3 times to guarantee the results. Based on pilot experimentation, 2, 4 and 6 mm lengths are chosen for testing. The specified lengths are produced using sieves attached to the grinding machine. The fibers and polymer (either virgin or recycled) are mixed using a mixer then the mix is fed into the single screw extruder.

![Figure 9: The extrusion machine](image)

Processing was carried out using the extrusion machine manufactured at the AUC labs shown in figure (9). The process starts by placing the mix of polymer and treated fiber in the hopper of the extrusion machine as shown in figure (10). Temperatures were set at 120 C for the first heater and 150 C for the second heater in the extruder as shown in figure (11). The first heater used to heat the mix to a desired temperature and properly melt it while the second heater ensures the flow of melted plastic with the additives before extrusion of the paste from the die. The heaters are insulated and covered with glass wool to contain heat and minimize heat loss. The cooling section is located at the beginning of the extruder with the water inlet and outlet to guarantee there is no melting in the mixing or feeding chamber as shown in figure (12). The mix is placed slowly so as not to clog up the rotating screw that allows input into the machine. The resulted extrudates are obtained
within 17 minutes for 500 grams. These effects were discovered during pilot experiment, which were reflected when the temperature range of the extruder was increased to 135°C and 165°C respectively. The melt was overheated, liquefied, and then stuck around the screw as it turned with its rotation without flowing. This caused the process to stop as it prevented the flow to continue and jammed the whole process. The difference in temperature is due to start with a primary heating then increase it to the final one as not to cause a sudden increase in temperature within the mix and therefore caused an incremental pressure resulted in exploding the mix rather than flowing.

Figure 10: Feeding the mixture in the hopper

Figure 11: The two heaters used to produce extrudates
Figure 12: The cooling section

The paste is taken to a hydraulic press to be pressed as shown in figure (13). A custom made steel die with specific dimensions is used to satisfy the requirements of testing as shown in figure (14). Trimming and cutting processes are done to make the product ready for testing in accordance with the requirements of the testing standards mentioned in the mechanical testing section.

Figure 13: The hydraulic press used
3.3 Pilot experimentation

Design of experiment is the design of information-gathering exercises where variation in factors is present. The need for an organized framework for doing experiments is a necessity. This saves time, money, effort by providing valid results with minimum number of experiments. DOE has a crucial role in engineering design, development and improvement of manufacturing processes. It has two main tasks. The first is setting efficient experimental design points with minimum number of runs. The second task is analyzing the factors involved within the experiments and showing the most important
ones. DOE has three branches; experiments with dependent, independent and hybrid factors. Experiments with dependent factors are concerned with factors having certain levels that are interacting in an experiment and are independent on each other affecting the response in a certain way. Experiments with hybrid factors are a combination between independent and dependent factors. A strategically planned and executed experiment provides information about the effect on a response variable due to several factors. To perform an experimental design, first the factors are chosen, variables are chosen and then performance of phase I & II experiments takes place. The interest in this thesis is to obtain a natural fiber reinforced polymer with the best possible physical and mechanical properties using a simple and effective technology [31].

Pilot experiments are phase I experiments. It consists of running initial experiments to get more experience and knowledge about the factors included and determines the important ones to be investigated further and exclude the unimportant ones. In this work, the initial experiments were conducted whether to use natural or synthetic fibers. The results showed that natural fibers are preferred due to their cost and availability. These experiments are the key which gives guidelines for necessary manufacturing techniques. The first experimental settings were built based on this stage: where literature review gave the way for a manufacturing technology. Two methods of manufacturing were suggested: injection molding method and extrusion and compression molding method. Then a local market survey was conducted to check the availability of machines needed for those two technologies [22]. The pilot experiments were run in random patterns to estimate the general behavior of factors. One concern was the humidity that was found in natural fibers. It was concluded that the percentage of humidity in rice straw was minimal.
There are several stages after the pilot experiments. Prerequisite stage consisted of making assumptions to start the experiment. It was based on literature review. In this case sequential modification of manufacturing a mold was done leading to the final manufacturing technology which extrusion and compression molding method. The treatment of natural fiber with an alkali or acid was decided upon according to the testing results. Stage I main target was to check the effect of treatment on the natural fiber and the adequacy of the chosen technology via its applicability using virgin or recycled plastic. Virgin plastic was used first to block any effect that could be accompanied with recycled plastic. As a result, the three manufacturing steps; extrusion, heating and compression have proven efficiency and gave a feasible product. Stage 2 is the stage of performing the experiment. The changing factors investigated; that are independent on each other are the fiber length, time of treatment, concentration of acid or alkali used. Another following step is the use of plastic waste rather than virgin plastic. Polymer composites have been subjected to increasing interest, study, and utilization for some decades. However, the increase in environmental concern has pointed out how it is also necessary to reduce and rationalize the use of polymeric materials, not only due to their non biodegradability, but also because their production requires large amount of oil as raw material which is not renewable. The product suffered inhomogeneous filler distribution in the final product. Plastic and fiber were not distributed evenly. This problem was solved using a grinder for grinding rice straw and recycled LDPE then mixing in a mixer. The process variables were taken into consideration in this stage. The furnace temperature, the extruder temperature and speed were controlled to produce efficient results without burning, overheating or solidification.

SEM analysis is used in the development and application of engineering measurements and test methods to the determination of materials and system behavior. The scanning electron
microscope is an electron microscope that images the sample surface by scanning it with a high-energy beam of electrons. It uses electrons rather than light to form an image. The electrons interact with the atoms that make up the sample producing signals that contain information about the sample's surface topography, composition. It is also designed for direct studying of the surfaces of solid objects. Several samples were subjected to tensile loading up to fracture

3.4 Procedures

Fiber preparation (based on literature review)

- Soak fibers in water for minimum 3 hours to make less brittle and separate easily.
- Separate fibers by sieving.
- Acid and Alkali treatment: approximately 30 grams fibers (weighed dry) are soaked in (1% and 5% of the acid or alkali solution (by weight). The solution is heated and stirred on an electrical heater until the temperature reaches 150°C and is kept at this temperature for 2 hours. During this process the container with the fibers and acid or alkali solution is fully covered using Al-foil. The temperature is constantly measured using a thermocouple.
- Wash fibers thoroughly with water after the chemical treatment.
- Cut fibers to 2, 4, 6 mm long parts. Fibers are cut using a grinding machine after drying
- Divide fiber agglomerations manually into smaller agglomerations.
- Dry fibers in a dryer at 60°C for 24 hours.
- Put fibers in blender (in small-quantity groups) for 5-6 sec in order to separate the agglomerations into single fibers.
- Use a sieve to get rid of the separated matrix material.
• Fibers can be dried again for a short duration (1-2 hours) at 100°C to get rid of any moisture.

• Store fibers well to prevent moisture absorption.

Composite preparation

Low Density Polyethylene is used as the matrix. It is characterized by good toughness, resistance to chemicals, flexibility and clarity. It's an excellent material in electrical and chemical uses in low heat applications. LDPE has a high degree of short and long chain branching. The chains do not pack into the crystal structure increasing ductility.

• LDPE is grinded (either virgin or recycled)

• 2%, 5% and 6% concentrations by weight of rice straw fiber are mixed with shredded LDPE in a mixer

• The mix is placed in a mold producing flexural and tension samples

• The mold is put in a press at 1900KPa and 120°C

Figure 15: The grinder used for plastic & fiber
3.5 Rice straw treatment using chemicals

The choice to use an acid or alkali will depend on both the tensile and flexural tests together with the uses of this waste after treatment. The uses of the acid and alkali solution were investigated in order to decide which acid or alkali should be used to treat rice straw. Phosphoric acid and Sulphuric acid as an acid solution will be investigated as well as sodium hydroxide as an alkali to select the best treatment solution. The waste from phosphoric acid treatment is used to remove rust from surfaces of metals. It can be used as a rust converter with the help of direct application to rusted iron, or steel tools and other rusted surfaces. The phosphoric acid converts reddish-brown iron, that is, ferric oxide to black colored ferric phosphate. After treatment with phosphoric acid, the black ferric phosphate coating can be scrubbed off, thus, revealing fresh metal surface. Phosphoric acid is also used as an additive to acidify foods and beverages like various colas. One of the most important phosphoric acid uses is in medicine and dentistry. Phosphoric acid is used as an ingredient in over-the-counter anti-nausea medications which also happen to contain high levels of sugar. In dentistry, this acid is combined with zinc powder to form zinc phosphate, which is used as a dental cement. It is also used in orthodontics as an etching solution so as to clean and roughen the surface of teeth, before the placement of brackets and other dental appliances. This acid is also used in many teeth whitening solutions to eliminate plaque that may be present on the surface of teeth. Moreover, the manufacture of most commercial phosphate fertilizers begins with the production of phosphoric acid [32].

Sulfuric acid is one of the most important industrial chemicals. The major use of sulfuric acid is in the production of fertilizers. It is widely used in the manufacture of chemical as hydrochloric acid, nitric acid, sulfate salts, synthetic detergents, dyes and pigments, explosives, and drugs. It is used in petroleum refining to wash impurities out of gasoline and other refinery products.
The sodium hydroxide alkali is commonly used as mentioned in the literature review, but its use as a waste after treatment is minimal. It is disregarded for treatment as the other acids are used after treating the fibers complying with the cradle to cradle approach. Regarding the concentration of acid used, it ranges from 1%-5% in previous papers as mentioned in the literature review section. The effect of 1 % and 5% will be studied on tensile and flexural stress. The lowest effective concentration will be chosen to reduce cost and chemical used.

3.6 Rice straw treatment through carbonization

- Rice straw after treatment with the acid or alkali is placed in a crucible
- The crucible is put into a furnace with a heating rate of 5 Cº/ min
- Rice straw is heated in vacuum at a temperature of 400 ºC for 1 hour
- After 1 hour the electric furnace is automatically switched off
- The door of the electric furnace is not opened until cooling to room temperature had taken place

3.7 Mechanical Testing

There are several tests that can be selected for FRP. Two main important areas of testing were performed. Flexural and tensile stress were chosen, as they are the ultimate range that the product can sustain under severe usage conditions. In addition, they are of a major importance for many international codes related to construction and mechanical requirement.
3.7.1 Tensile stress

It is the maximum stress that a material can withstand while being stretched or pulled before necking, when the specimen's cross-section starts to significantly contract. It is found by performing a tensile test and recording the stress versus strain; the highest point of the stress-strain curve is the ultimate tensile stress. It is an intensive property; its value does not depend on the length of the test specimen. It is dependent on other factors, including preparation of the specimen, the presence of surface defects, and the temperature of the test environment and material.

The Instron machine used in the experimental procedure is 3300 Instron as shown in figure (16). Instron is a testing machine having properties of performing tensile, compression, flex, peel and cyclic type of testing. It is attached to an online computer and via computer software all the required orders are taken. It is available at the AUC testing labs and was utilized to perform flexural and tensile tests. The software utilized; Instron BLuehill Lite is designed to run Instron’s Model 3300 Material Testing Systems. First the two edges of the machine consist of the support span, where the grips are attached to the load ram and the specimen was concentrated between the support edges. The machine is turned on with the computer. The next step was to adjust the computer software settings. The computer settings required three major set of data regarding the specimen dimensions, feed rate control and output type. After adjusting the computer, software settings, the machine was ordered to start the test and the ram moved downwards applying the load. After the completion of the test, the ram returns to its initial positioning and a new specimen was added. The output of the test consisted of a stress-strain diagram and a table containing the details of each specimen.
Samples were cut using the Isomet cutting machine or a simple saw. Sample dimensions were measured using a digital Vernier at 3 locations and an average was taken. Tension samples (ASTM D3039/D 3039M – 00) [27]:

- Width = 20 mm
- Length = 100 mm
- Thickness = approx. 4.5 – 6.5 mm
- Grip = 20 mm
- Crosshead speed = 5 mm/min

Modulus of elasticity is also calculated, it is the mathematical description of an object. It is the tendency of the substance to be deformed elastically when a force is applied to it. Young's modulus ($E$) describes the tendency of an object to deform along an axis when opposing forces are applied along that axis; it is defined as the ratio of tensile stress to tensile strain.

Figure 16: Instron Universal testing machine with tension grips
3.7.2 Flexural stress

The flexural test is done in several methods: 3-point loading, 4-point load or uniform load. In all cases, the specimen is supported with 2 edges and a load is applied with a known feed rate. The difference between the methods is mainly based on the number of load noses applied; that could be one or two, the distance between these noses; the maximum bending moment. In the case of 3-point loading, the specimen is loaded with one nose in the middle of the specimen support span; the distance between the 2 support edges. Therefore the maximum axial fiber stress is positioned directly under the loading nose. While in the 4-point loading the maximum axial fiber stress is uniformly distributed between the loading noses. The uniformly distributed loading is performed using one of the 3-point or 4-point load. The uniform load is calculated using standard equations this method is not commonly used due to its technical difficulties.

Flexural stress is the material's ability to resist deformation under load. The transverse bending test is most frequently employed, in which a rod specimen having either a circular or rectangular cross-section is bent until fracture using a three point flexural test technique. The flexural stress represents the highest stress experienced within the material at its moment of rupture. It is measured in terms of stress. The equation for a rectangular sample under a load in a three-point bending: \( \sigma = \frac{3FL}{2bd^2} \) where \( F \) is the load (force) at the fracture point, \( L \) is the length of the support span, \( b \) is width, \( d \) is thickness [33].

Bending samples (ASTM D790 – 03) [27]:

- Width = 20 mm
- Thickness = approx. 4.5 – 6.5 mm
- Span = 16 x thickness
- Crosshead speed = 2 mm/min
Critical Fiber length

There is an important issue that was considered concerning the critical fiber length which is calculated according to an equation $L_{critical} = \frac{\sigma \text{ fracture} \times D}{2 \times \zeta}$ where $\sigma$ is the stress, $D$ is the diameter of fiber and $\zeta$ shear stress. The value for shear stress was obtained for the matrix (LDPE) 200 MPa, the diameter of the fiber is 20 µm, $\sigma$ flexural $= 4$ Mpa, $\sigma$ tensile $= 9$ Mpa. The critical fiber length using $\sigma$ flexural is 0.02mm, while the critical length using $\sigma$ tensile is 0.045. the fiber length that will be used is 2, 4, 6 mm. This procedure ensures that the chosen fiber length according based on the literature review can be safely used.
Chapter 4

Results & Discussion

4.1 Introduction

The polymer used in the first experimentation process is LDPE due to its economical and technical advantages. The second experimentation is done using recycled LDPE of thermoplastic nature. The third experiment is done using rejects. The virgin, recycled LDPE and rejects produced need to be grinded then mixed with the treated rice straw fibers. The mixture is placed in the extruder to produce the desired paste. The paste is placed in a manufactured die producing flexural and tensile samples. The shape of different kinds of samples before and after for both flexural and tensile tests are shown in figure (18) & (19). Flexural test is one of the mechanical tests that are used to measure the material behavior when subjected to simple beam loading which is known as the transverse beam test. It is the maximum fiber stress that could be developed before breaking. The specimen in this test is supported with 2 ends and a load is to be applied with a known feed rate. Maximum fiber stress and strain are calculated and plotted in a stress-strain diagram. These results are of a major importance for the international code council evaluation service acceptance criteria and other code depending on the type of application.
4.2 Effect of fiber reinforcement on virgin LDPE using flexural test

Fibers are considered the main element in resisting the load in the fiber reinforced composites, since the physical and mechanical properties of the composite material depends mainly on the type, length, and properties of the fibers used as well as it depends on the volume fractions of and the direction and orientation of the fibers in the matrix. Also, it depends on the interfacial bond between the fibers and the polymeric matrix.
Natural fibers, as reinforcement, have recently attracted the attention of researchers because of their advantages over other established materials. They are environmentally friendly, fully biodegradable, abundantly available, renewable and cheap and have low density. Plant fibers are light compared to glass, carbon and aramid fibers. The biodegradability of plant fibers can contribute to a healthy ecosystem while their low cost and high performance fulfils the economic interest of industry. The control sample is a flexural stress-strain diagram using virgin LDPE with 0% rice straw.

4.2.1 Effect of chemical concentration & fiber concentration

The effect of 0, 2, 5 and 6% (by weight) of treated fiber concentration using 1 and 5% chemical concentration with 2 mm fiber length on the reinforcement of VLDPE will be investigated in this section using flexural test.

Reinforcement with treated fiber using NaOH:

Three samples for each condition were tested in accordance with ASTM D 790-03 to determine the flexural stress of FRP of 0, 2, 5, and 6% rice straw of length 2 mm as shown in figures (20). A standard fixture is used that has two equal concentrated points of load application spaced equidistant between the supports. A constant head speed of 2 mm/min was used to allow for failure anywhere between 5-10 minutes. The increase in fiber content showed an improvement in flexural stress till 5% fiber and a decrease in flexural stress above this fiber content. Based on the literature review, samples of 1 and 5% concentration of Na OH were chosen. The 5% concentration of the alkali showed no significant change in flexural stress than 1% not more than 0.5 MPa as shown in figure (21). The most suitable sample is 1% concentration of the alkali and 5% rice straw of length 2 mm producing 3.5 MPA as shown in figure (20). The use of low concentration promotes the cleaner production technique with the reduction of chemicals at the source as well as less cost.
Reinforcement using treated fiber with H$_2$SO$_4$:

The same procedure for sodium hydroxide treatment is done using sulphuric acid treatment for each test as shown in figure (22). The increase in fiber content from 2 & 5% showed an improvement in flexural stress and a decrease in flexural stress at 6 % fiber. The concentration of the acid showed no change in flexural stress. The most suitable
sample is 5% rice straw of 2mm length treated with 1% concentration of H₂SO₄ as shown in figure (23).

![Figure 22: Representative variation of flexural stress for different fiber conc. with 2 mm length](image)

**Figure 22: Representative variation of flexural stress for different fiber conc. with 2 mm length**

![Figure 23: Representative variation of flexural stress with different conc. of H₂SO₄](image)

**Figure 23: Representative variation of flexural stress with different conc. of H₂SO₄**

**Reinforcement using treated fiber with H₃PO₄:**

The same procedure for previous treatment is done using phosphoric acid with several samples repeated for each test as shown in figures (24). The increase in fiber content from
2.5 showed an improvement in flexural stress and a decrease of flexural stress at 6% fiber. The concentration of the acid showed slight change in flexural stress as shown in figure(25). The most suitable sample is 5% rice straw of 2mm length treated with 1% concentration of H₃PO₄. The flexural stress values of NaOH treated fiber and the H₃PO₄ treated fibers are higher than the H₂SO₄ treated fibers as shown in figure (26). It can be concluded that the most suitable treatment is the one with the H₃PO₄ acid rather than NaOH treatment where the waste generated from the fiber treatment can be utilized as a fertilizer for reclamation. The chemical concentration of 5% was mentioned in the literature review. The 1% chemical was experimented; it showed almost the same values of flexural stress so it is better to use the lowest possible chemical concentration.

Figure 24: Representative variation of flexural stress for different conc. of fiber with 2 mm length
Figure 25: Representative variation of flexural stress for different conc. of $\text{H}_3\text{PO}_4$

Figure 26: Effect of treatment media on flexural stress using 1% chemical conc. & 5% rice straw
4.2.2. Effect of natural fiber length and fiber concentration

The effect of 5% of treated fiber concentration using 1% chemical concentration with 2, 4, and 6 mm fiber length on the reinforcement of VLDPE will be investigated in this section using flexural test.

![Figure 27: Effect of fiber length on flexural stress using 1% chemical conc. & 5% fiber conc.](image)

Flexural stresses were decreased for composites with 4 and 6 mm length compared to the 2 mm fiber reinforced composites as shown in figure (27). The probable reason is that a long length of the fiber is not compatible with the matrix properly thus causing distortion. Moreover, fibers may be folded and there is no bonding between the folded and unfolded portion of fiber which resulted in a lower stress. Fiber entanglement may also contribute to reduce the stress. The trend to increase the fiber content to 6 % and increase the fiber length decreased flexural stress as observed in figure (28). The increase in fiber length to 6mm as reinforcement in the composites, flexural stress was reduced. The incorporation of fibers into the polymer led to poor dispersion of fibers. Improper adhesion hinders the flexural stress as the fiber percentage and length increases. It is concluded from the
previous tests of acids and alkalis that the suitable acid is the phosphoric acid with a concentration of 1% and 5% fiber content.

![Figure 28: effect of increasing fiber conc. & fiber length on flexural stress](image)

4.3 Effect of fiber reinforcement on virgin LDPE using tensile test

The effect of 0, 2, 5, and 6% (by weight) of treated fiber concentration using 1 and 5% chemical concentration with 2 mm fiber length on the reinforcement of VLDPE will be investigated in this section using tensile test.

4.3.1 Effect chemical concentration & fiber concentration

Reinforcement with treated fiber using NaOH:

Three samples were tested for each condition in accordance with ASTM D 3039 to determine the tensile stress of FR P of 2,5,6% rice straw of length 2 mm as shown in figures (29). The increase in fiber content showed an improvement in tensile stress till 5% fiber and a decrease in stress above this fiber content. Samples of 1 and 5% concentration of Na OH were also tested as shown in figure (30). The 5% concentration of the alkali
showed no significant change in tensile stress than 1%. The most suitable sample is 1% concentration of the alkali and 5% rice straw of length 2mm. The use of low concentration promotes the cleaner production technique with the reduction of chemicals at the source as well as less cost.

Figure 29: Representative variation of tensile stress for different conc. of rice straw with 2 mm length
Reinforcement using treated fiber with $\text{H}_2\text{SO}_4$:

Three samples for each condition were tested in accordance with ASTM D 3039 to determine the tensile stress of FRP of 0, 2.5, and 6% rice straw of length 2 mm as shown in figures (31). The increase in fiber content showed an improvement in tensile stress till 5% fiber and a decrease in flexural stress above this fiber content. Samples of 1 and 5% concentration were also tested as shown in figure (32). The 5% concentration of the acid showed no significant change in tensile stress than 1 %. The most suitable sample is 1 % concentration of the acid and 5 % rice straw of length 2 mm . The use of low concentration
promotes the cleaner production technique with the reduction of chemicals at the source as well as less cost.

**Figure 31:** Representative variation of tensile stress for different conc. of fiber with 2 mm length

![Graph showing tensile stress for different concentrations of fiber with 2 mm length](image)

**Figure 32:** Representative variation of tensile stress with different H₂SO₄ conc.

![Graph showing tensile stress with different H₂SO₄ concentrations](image)

**Reinforcement using treated fiber with H₃PO₄:**

Several samples were tested in accordance with ASTM D 3039 to determine the tensile stress of FRP of 2, 5, 6% rice straw of length 2 mm as shown in figures (33). The increase in
fiber content showed an improvement in tensile stress till 5% fiber and a decrease in flexural stress above this fiber content. Samples of 1 and 5% concentration of H$_3$PO$_4$ were also tested as shown in figure (34). The 5% concentration of the acid showed no significant change in tensile stress than 1 %. The most suitable sample is 1 % concentration of the acid and 5 % rice straw of length 2mm. The use of low concentration promotes the cleaner production technique with the reduction of chemicals at the source as well as less cost. It is concluded that phosphoric acid treatment has the highest tensile stress compared to the other acid and alkali as shown in figure (35).

Figure 33: Representative variation of tensile stress for different fiber conc. with 2 mm length
4.3.2 Effect of natural fiber length and fiber concentration:

The effect of 5 and 6 % of treated fiber concentration using 1 % chemical concentration with 2, 4 and 6 mm fiber length on the reinforcement of VLDPE will be investigated in this section using tensile test. The use of 1 % chemical concentration as it obtains almost the same stress as using 5 % chemical concentration.
Tensile stress for composites of fiber length 4 and 6 mm decreased, compared to 2 mm fiber reinforced composites as shown in figure (36). The improper bonding occurs between the fibers and the matrix due to increase in fiber length. Moreover, fibers may be folded and there is no bonding between the folded and unfolded portion of fiber which resulted in a lower stress. Fiber entanglement may also contribute to reduce the stress. The trend of increase the fiber content to 6 % with an increase of fiber length, caused a decrease of tensile stress as observed in figure (37). The incorporation of fibers into the polymer led to poor dispersion of fibers. Improper adhesion hinders the flexural stress as the fiber percentage and length increases. It is concluded from the previous tests of acids and alkalis that the suitable acid is the phosphoric acid with a concentration of 1% and 5% fiber content. The lowest tensile stress of the rice straw with length 6mm was observed in figure (37).
4.4 Effect of fiber reinforcement on recycled LDPE using flexural test

Based on the findings of VLDPE and the results of the literature survey, RLDPE can be used as the matrix in the fiber reinforced composite. The effect of chemical concentration, fiber concentration and effect of using treated fiber through carbonization will be investigated in this section. The control is a flexural stress- strain diagram using recycled LDPE with 0 % fibers. It is used for the sake of comparison with virgin LDPE.
4.4.1 Effect of chemical concentration

The effect of 1 and 5% chemical concentration with 2 mm fiber length on the reinforcement of RLDPE will be investigated using flexural test. According to figure () the two concentrations almost obtained the same result so the 1% will be used in order to avoid the use of unneeded chemicals.

![Figure 38: Representative variation of flexural stress for different chemical conc. Of H₃ PO₄](image)

4.4.2 Effect of fiber concentration

The same testing procedure for virgin LDPE is done using three samples of the recycled polymer as shown in figures (39). The effect of fiber content 0, 2, 5 and 6% is tested with 2 mm length. The most suitable sample is 5% rice straw of 2mm length treated with 1% concentration of H₃ PO₄ as shown in the previous tests. The increase in fiber content lowered the flexural stress.
4.4.3 Effect of fiber length

The effect of 5% of treated fiber concentration using 1% chemical concentration with 2, 4 and 6 mm fiber length on the reinforcement of RLDPE will be investigated using flexural test. The use of 1% chemical concentration as it obtains almost the same stress as using 5% chemical concentration. Figure (40) showed the effect of increasing fiber length leading to a decrease in flexural stress. At high fiber volume fractions, the material properties show greater variability due to fiber clumping. Also the fiber length decreases with increasing fiber content and this reduction in fiber length then reduces fiber reinforcing efficiency due to the increased fiber–fiber interaction and fiber–equipment wall contact. Moreover the increase in fiber content causes the formation of agglomerates acting as a stress focus and, therefore, as a propagator of cracks, resulting in the greater tenacity of the composites.
4.4.4 Effect of treatment through carbonization

The fibers are treated through carbonization. The effect of treated carbonized fiber is investigated using 2 and 5 % fiber content. The sample with the treated, carbonized -5% rice straw has higher flexural stress according to figure (41).
4.5 Effect of fiber reinforcement on recycled LDPE using tensile test

Based on the findings of VLDPE and the results of the literature survey, RLDPE can be used as the matrix in the fiber reinforced composite. The effect of chemical concentration, fiber concentration and effect of using treated fiber through carbonization will be investigated in this section. The control is a flexural stress-strain diagram using recycled LDPE with 0 % fibers.

4.5.1 Effect of chemical concentration

The effect of 1 and 5 % chemical concentration with 2 mm fiber length on the reinforcement of RLDPE will be investigated using tensile test. According to figure (42) the two concentrations almost obtained the same result so the 1 % will be used in order to avoid the use of unneeded chemicals.
4.5.2 Effect of increasing fiber concentration

The same testing procedure for virgin LDPE is done with several samples of the recycled polymer as shown in figures (43). The effect of fiber content 2, 5 and 6% is tested with 2 mm length. The most suitable sample is 5% rice straw of 2mm length treated with 1% concentration of H₃PO₄ as shown in the previous tests. The increase in fiber content lowered the tensile stress.
4.5.3 Effect of fiber length

The effect of 5% of treated fiber concentration using 1% chemical concentration with 2, 4 and 6 mm fiber length on the reinforcement of RLDPE will be investigated using tensile test. The use of 1% chemical concentration as it obtains almost the same stress as using 5% chemical concentration. Figure (44) showed the effect of increasing fiber length leading to a decrease in flexural stress.
4.5.4 Effect of treatment through carbonization

The fibers are treated through carbonization. The effect of treated carbonized fiber is investigated using 2 and 5% fiber content. The sample with the treated, carbonized 5% rice straw has higher tensile stress according to figure (45).

![Figure 44: Representative variation of tensile stress for different fiber length](image)

4.6 Micro structure analysis

The control is the virgin LDPE presented in figure (82). The fracture surfaces were examined in a scanning electron microscope and are shown in Figure (83). It is observed that in each case the cells elongate under tensile stress and acquire a permanent deformation. The rice straw fibers are clear at the tip of the fracture surface as shown in figure (84). The fiber-matrix dispersion is reflected in figure (85) in several spots as the fibers are well embedded in the polymer. A close up shot is taken in figure (86) to show the interface between the polymer and the fiber. The shape of fracture differs with the type

![Figure 45: Representative variation of tensile stress for different treated, carbonized fiber conc.](image)
of polymer used either recycled or virgin. There is a degree of plasticity and ductility in VLDPE as shown in figure (87). The degree of ductility increases with the use of recycled LDPE. The necking is seen in the specimen and elongation persists for quite a while showing high plasticity as shown in figure (88). Figure (89) shows the structure of the recycled sample is more ductile but there are some agglomerations which are not found in the control sample in figure (82) due to the inhomogeneous mixing while mixing fiber and polymer during extruding.

From this micro analysis it is clear that interfacial stress between the rice straw and LDPE matrix is the most indispensable factor for achieving good fiber reinforcement. The interface acts as a “binder” and transfers the load between the matrix and the reinforcing fibers. Mechanical stress of the composites levels decrease at high fiber loading. This behavior can be explained on the basis that at higher fiber loading the fiber-fiber contact dominates over the resin matrix-fiber contact, which decreases the mechanical properties.

Figure 46: SEM micrograph of virgin LDPE
Figure 47: SEM micrograph showing fracture surface of virgin FRP

Figure 48: SEM micrograph showing fracture surface of virgin FRP -2

Figure 49: SEM micrograph showing fracture surface of virgin FRP-3
Figure 50: SEM micrograph showing the polymer-fiber pull-out of virgin FRP

Figure 51: Shape of fracture of virgin FRP sample

Figure 52: Shape of fracture of recycled FRP sample
Figure 53: SEM micrograph showing smooth and ductile surface of recycled FRP
Chapter 5

Conclusions

Based on the analysis regarding the virgin LDPE, the reinforcement with rice straw as a natural fiber improved the flexural & tensile properties of the polymer. Several chemical treatments of natural fibers were tested. The most suitable chemical was the phosphoric acid with 1 % chemical concentration due to obtaining the highest values of tensile and flexural stress, rather than 5 % chemical concentration that was documented in the literature survey. Moreover, the waste generated from the chemical treatment using phosphoric acid can be utilized as a fertilizer component rather than sodium hydroxide and sulphuric acid. The fiber used was rice straw with fiber concentration of 5 % as it showed the highest mechanical properties. This is shown in table 3 with all the values that will be discussed further. This percentage was recommended through literature review. Increasing the fiber concentration to 6 % in the experimentation stage lowered the flexural and tensile stresses. The values of flexural stress decreased from 3.5 to 2.9 MPa, while the values of tensile stresses were reduced from 9 to 5 MPa. Therefore no further increase of fiber concentration was tested. The decrease of fiber concentration to 2 % rice straw was also tested. The values obtained were 6 MPA for tensile stress and 1.5 for flexural stress as shown in table 3. This means that decreasing the fiber concentration to 2 % obtained lower results in both tests by 3 times. This concluded that 5% fiber concentration was the best possible fiber concentration and will be used for the rest of the experimental procedure. In addition, increasing the fiber length from 2 mm length to 4 and 6 mm length produced lower flexural and tensile stresses by 6 times. The variation in fiber length was tested with
the increase of fiber concentration to make sure that 5 % fiber concentration produced the highest tensile and flexural stresses with an increase 3-8 times more than virgin LDPE.

The mechanical properties of recycled LDPE are lower than the mechanical properties of virgin LDPE. However the reinforcement with chemically treated rice straw, the properties of the material increase by 3 times. That enables it to be used for several applications due to durability, environmental friendliness, ease of processing, stress, toughness, flexibility, ease of sealing and barrier to moisture. The effect of increasing the fiber concentration to 6 % and decreasing it to 2 % was tested to make sure that increasing the fiber concentration lowered both tensile and flexural stress and lowering it to 2 % also lowered both stresses by 3 times. All the values mentioned are highlighted in table 3. The reason for retesting these parameters is the transfer from virgin to recycled LDPE where there might be a change in properties. The effect of increasing fiber length was studied and it agreed with the virgin LDPE. Moreover, the treatment of rice straw through carbonization was addressed as the mechanical properties of recycled LDPE reinforced with rice straw were low compared to virgin composite by 5 times, but still higher than the recycled LDPE. This treatment contributed to the formation of fibers with higher values of surface area and micro porosity. This enabled a high adsorption capacity for micro pollutants. This carbonization enhanced the environmentally utilization of undesirable cellulosic wastes and the carbonized rice straw with the recycled LDPE resulted in higher values of tensile stress by 6 times compared to the recycled LDPE reinforced with chemically treated rice straw. There was an improvement in tensile stress from 3.5 to 9 MPa after using the carbonized rice straw with a concentration of 5 %. This value showed a great improvement reaching the value of the virgin LDPE reinforced with chemically treated fiber. The effect of treatment through carbonization in flexural stress was the same value compared to recycled polymer and reinforced with chemically treated fiber.
Table 3: Effect of different parameters of rice straw on flexural and tensile stress

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<th>Polymer</th>
<th>Chemical Treatment</th>
<th>Treatment conc.</th>
<th>Fiber conc.</th>
<th>Fiber length</th>
<th>Tensile stress (MPa)</th>
<th>Flexural stress (MPa)</th>
<th>Youngs Modulus (MPa)</th>
<th>Modulus of Toughness (MPa)</th>
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**Recommendations**

Exploring FRP hybrid systems is a further step to produce composites with improved mechanical properties. FRP Hybrid systems is using more than one type of fiber in the production of FRP products, or the use of different FRP products (plates, laminates, bars, sections) together or with another materials. Hybrid systems are used to utilize the physical and mechanical properties of each of the used materials and to offset the disadvantages and deficiency of the others, in order achieve design requirements and economics. These hybrid systems requires extensive familiarity with the physical and mechanical properties of the used materials besides its chemical composition in order to avoid any incompatibility and chemical reactions that could affect the long term durability. This study requires full knowledge and experience with the different materials used and systems to achieve the structural performance required.

The properties of reinforced fibers depend on the type of fibers, orientation of fibers, direction either unidirectional or two directions, manufactured source, and the processing methods. The fibers are arranged through mono direction inside the composite, results in maximum tensile strength and maximum modulus of elasticity in the fibers axis direction. In case of arranging the fibers in bidirectional, it will show different strength in coordination with the different angles. The mechanical properties in any of the directions are proportional to the fiber volume fraction in that direction. One way is gathering fibers together in the form of laminates and several layers of laminates could be glued to each other to reach the required thickness of the fiber reinforced laminates thus producing higher strength. The fibers orientation and direction in each laminate could be controlled during the preparation and production process, also the sequence of adding these laminates to each other could be controlled [34].

Another future recommendation a detailed economic analysis should be performed including the price of the product after calculating the fixed cost including the cost of equipment, machinery, land and the operating cost including cost of raw material, labor and utilities. Another improvement to the manufacturing process is the mixing process. The use of another mixer could be investigated to avoid the inhomogeneous mixture that causes agglomerations especially with the increased length of fibers.
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