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Cellulose Extraction from Sisal Fibers and Thermo-Chemical Characterization for Sustainable Industrial Applications

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ABSTRACT

The research on cellulose extraction from sisal fibers and its application in industry is an important field of study, especially with regard to the creation of environmentally friendly materials. The aim of this particular study was to utilize natural fiber waste and promote sustainability by extracting cellulose from sisal fibers. Several analytical methods, such as EDX, TGA, DSC, and tensile testing, were used to evaluate the properties of the extracted cellulose fibers. The results show that the extracted cellulose was thermally stable and had good mechanical properties, making it suitable for various industrial purposes. The use of natural fiber waste as an alternative to synthetic materials is receiving increasing attention due to its potential to reduce the environmental impact and promote circular economy practices. This study contributes to the growing body of research on sustainable materials and highlights the potential of using natural fiber waste in industry. Additionally, it provides insights into the potential use of sisal fibers in other areas, such as biodegradable packaging materials, textiles, and medical applications. The development of sustainable materials is crucial in addressing environmental challenges and promoting sustainable development, and this study provides valuable insights into the use of natural fibers as an environmentally friendly alternative to synthetic materials.

Keywords: Biomass; Biodegradable; Cellulose; Natural; Sisal; Sustainable

1. Introduction

Cellulose, a natural polymer found in the cell walls of plants, is the most abundant biopolymer in the world. Its unique properties make it a valuable material in various sectors such as food, cosmetics, pharmaceuticals and textiles. However, the main source of cellulose for industrial purposes comes from the extraction of wood pulp using chemical or mechanical methods

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applied to plants. This manufacturing process is associated with significant energy and water consumption, as well as significant environmental impacts. Therefore, it is imperative to explore alternative sources of cellulose that are more sustainable and environmentally friendly [1-5]. Natural fibers from these alternative sources could provide greener solutions and reduce the overall environmental footprint associated with cellulose production. This search for sustainable sources is critical not only to diversify cellulose supplies, but also to mitigate negative impacts on the ecosystem. Research in this area is essential to progress towards more responsible industrial practices that respect our planet.

Natural fibers, such as sisal, jute and hemp, are a potential alternative source of cellulose that can be obtained from plant waste. These fibers are renewable, biodegradable and have excellent mechanical properties, making them suitable for various applications. Sisal, in particular, is a tropical plant native to Mexico and is cultivated in many countries, including Brazil, Tanzania and Kenya. The sisal plant produces long, strong fibers that are commonly used for rope, twine and carpet backing [6-8].

The use of natural fiber waste as an alternative to synthetic materials is receiving increased attention due to its potential to reduce environmental impact and promote circular economy practices. By using sisal fibers as a source of cellulose, this study contributes to the growing body of research on sustainable materials and highlights the potential of using natural fiber waste in industry. In addition, the study provides insights into the potential use of sisal fibers in other areas such as biodegradable packaging, textiles, papermaking, nanocomposites, and medical applications [9-13].

Investigating the process of extracting cellulose from sisal fibers is very promising, especially in terms of producing sustainable products. The objective of this study was to separate cellulose from sisal fibers and evaluate its suitability for industrial applications. The sisal fibers were subjected to a series of chemical and mechanical treatments to remove contaminants and separate the cellulose fibers. A variety of analytical techniques including tensile testing, TGA, DSC and EDX were used to evaluate the properties of the isolated cellulose fibers. Energy dispersive X-ray spectroscopy, or EDX, is a method for determining the chemical composition of a sample. In this investigation, EDX was used to determine the elemental composition of sisal and extracted cellulose fibers. The thermal stability of the isolated cellulose fibers was evaluated using differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA). The mechanical properties of the extracted cellulose fibers, such as tensile strength, Young's modulus, and elongation at break, were determined by tensile testing.

The results of the study showed that the extracted cellulose fibers had a high cellulose content (94.9%) and were thermally stable up to 250°C. Tensile test results showed that the extracted cellulose fibers had good mechanical properties, with a tensile strength of 166 MPa, Young's modulus of 7.87 GPa, and elongation at a break of 2.75%. These properties make the extracted cellulose fibers suitable for various industrial applications, including the production of biodegradable packaging materials, textiles, and medical products.

The development of sustainable materials is critical to addressing environmental challenges and promoting sustainable development. This study provides valuable insights into the use of natural fibers as an environmentally friendly alternative to synthetic materials. By using sisal fibers, a type of natural fiber waste, to extract cellulose, this study contributes to the development of sustainable materials and promotes circular economy practices. Overall, the study highlights the importance of exploring alternative sources of cellulose and developing sustainable materials to address environmental challenges and promote sustainable development.

2. Methods and materials

2.1 Materials and chemicals

Sisal is a natural plant fiber obtained from the leaves of the Agave sisalana plant. Although the plant is native to Mexico, it is also grown in a number of other countries, including Tunisia. The sisal extraction process begins with peeling, an operation that separates the fibers from the unusable parts of the leaf.

Once the fibers are extracted, they go through another process to obtain the cellulose. At this stage, chemicals such as sodium hydroxide (caustic soda), acetic acid and sodium chlorite are used. These chemicals play a critical role in breaking down lignin, a non-cellulose component present in sisal fibers. The lignin is removed to isolate pure cellulose, resulting in sisal fibers that are stronger and more suitable for a variety of applications.

2.2 Cellulose extraction

The materials and chemicals used in this work to extract cellulose from sisal fibers were purchased from a local source and included sisal fibers, sodium hydroxide (NaOH), sodium chlorite (NaClO2), and glacial acetic acid. Sisal fibers are derived from the sisal plant and are widely used in rope, twine, and carpet backing. NaOH, a strong base, has been used to remove impurities and hemicellulose from sisal fibers at a concentration of 10% (w/v). NaClO2, a bleaching agent, has been used to brighten sisal fibers at a concentration of 1.7% (w/v) and to remove lignin from lignocellulosic fibers. CH3COOH, a weak acid, was used to modify the pH of the bleached sisal fibers at a concentration of 1% according to [14-16]. All compounds used in this investigation were purchased from a commercial supplier and were analytical grade. The methods used in this work to extract cellulose from sisal fibers followed known procedures and were carefully carried out to obtain reliable results.

2.3 Characterisation

2.3.1 Mechanical proprieties

To analyze the mechanical properties of native sisal fibers and cellulose derived from sisal fibers, a tensile test was performed using a Shimadzu machine. This machine is widely used in materials testing and is capable of measuring the tensile strength and Young's modulus of various materials. During the test, the natural sisal fibers and the cellulose extracted from the fibers were subjected to increasing tensile forces until they broke. Tensile strength, also known as ultimate tensile strength, is the maximum stress a material can withstand before breaking. It is calculated by dividing the maximum force required to break the material by the cross-sectional area of the specimen. The formula used to calculate tensile strength is

$$Tensile\ Strength = Maximum\ Force / Cross\ Sectional\ Area,$$

$$\sigma = \frac{F_{Max}}{S}. \tag{2.1}$$

Young’s modulus, also called modulus of elasticity, is an indicator of the stiffness of a material. This property is determined by the stress-strain ratio in a material subjected to tensile or compressive forces within its elastic limit. The mathematical expression for calculating Young’s modulus is described as follows:

$$Young's\ modulus = Stress / Strain,$$

$$E = \frac{\sigma}{\varepsilon}, \tag{2.2}$$

where $\varepsilon = \frac{\Delta L}{L}$. (2.3)

2.3.2 Chemical analysis by EDX

EDX analysis is a powerful analytical technique that is widely used to determine the elemental composition of a given sample. The method, which stands for Energy Dispersive X-ray Spectroscopy, is based on the principle of measuring the energy of X-rays emitted by the sample. These X-rays are produced when the sample is bombarded with high-energy electrons, causing the atoms in the sample to emit characteristic X-rays that are unique to each element.

In this particular case, EDX analysis was used to examine the elemental composition of cellulose extracted from sisal fibers and compare it to the elemental composition of natural sisal fibers. The goal was to identify any differences between the two samples and gain a better understanding of their respective properties [17].

By analyzing the X-rays emitted by the samples, EDX analysis was able to determine the different elements present in the cellulose and natural sisal fiber samples, as well as their relative abundances. This information allowed the researchers to compare the two samples and identify any differences in their elemental composition.

2.3.3 Thermal analysis

Thermogravimetric Analysis (TGA) and Differential Scanning Calorimetry (DSC) are two common techniques used to evaluate the thermal stability properties of natural sisal fiber and its derived cellulose.

TGA is a method that observes changes in the weight of a sample as it undergoes controlled heating or cooling in a specified environment. This technique is particularly useful for evaluating the thermal stability of materials because it can detect the onset of decomposition or degradation. When applied to natural sisal fiber and cellulose, TGA provides a complete understanding of their decomposition temperature. This information is essential for assessing the suitability of these materials for various applications, providing insight into their thermal resilience and potential use in specific contexts [18-20].

In essence, TGA is a critical tool for characterizing the thermal behavior of natural sisal fiber and cellulose, providing valuable data for making informed decisions regarding their applicability in various fields.

Conversely, DSC, measures the heat flow into and out of a sample as it is subjected to a controlled temperature program. This technique can provide information on the thermal properties of materials, such as their melting points, glass transition temperatures, and specific heat capacities. In the case of natural sisal fiber and cellulose, DSC can be used to determine their thermal behavior, such as their melting points, glass transition temperatures, and other related properties, using a state-of-the-art "TGA/TDA 1600°C/DSC 800°C thermobalance" under controlled conditions. Samples were heated at a rate of 10°C/min in a nitrogen environment, and the temperature range for testing was set between 25 and 800°C.

2.3.4 Physical proprieties

The physical properties of natural sisal fiber and cellulose extracted from them can provide valuable insights into their potential uses in various industries. Two commonly measured physical properties are absorption rate and density.

Absorption rate is an important property for materials that may come into contact with liquids or gases. The ability of natural sisal fiber and cellulose to absorb water, for example, can impact their suitability for use in applications such as packaging, textiles, or paper products. The absorption rate of these materials can be measured using various techniques, such as water immersion or contact angle measurement.

Density, on the other hand, refers to the mass per unit volume of a material. Natural sisal fiber and cellulose are lightweight materials, and their density can vary depending on factors such as the extraction method and processing techniques. Understanding the density of these materials can be useful for determining their suitability for various applications, such as in lightweight composites or insulation materials.

Overall, the physical properties of natural sisal fiber and cellulose extracted from them can provide important information on their potential applications in various industries. By understanding the absorption rate and density of these materials, researchers and manufacturers can make informed decisions about their suitability for different uses and work towards improving their environmental sustainability.

3. Results

3.1 Mechanical and structural proprieties

As shown in Table 1, sisal fibers have a tensile strength of 125 MPa, while the cellulose extracted from them has a higher tensile strength of 530 MPa. The Young's modulus of sisal fibers is approximately 62 GPa, which is lower than the Young's modulus of cellulose fibers, which is approximately 131 GPa. However, sisal fibers have a comparable elongation at break of 3-5% for 5 specimens, while cellulose fibers have an elongation at break of approximately 2-5%. Sisal fibers have a wider diameter range, from 80 to 220 mm, while cellulose fibers have a diameter range of 40 to 75 μm.

We can say that cellulose fibers have several advantages over natural fibers based on their properties. First, cellulose fibers have a higher tensile strength and Young's modulus than many natural fibers, such as sisal or cotton fibers. This makes cellulose fibers stronger and stiffer, which is important in applications where high strength and stiffness are required. Second, cellulose fibers have a more uniform microstructure than many natural fibers, which allows for more consistent mechanical and structural properties. This makes cellulose fibers more predictable and easier to use in manufacturing processes. Third, cellulose fibers have a higher degree of purity than many natural fibers. This means that cellulose fibers are less likely to contain impurities that can affect their properties or cause degradation over time. Finally, cellulose fibers have excellent chemical resistance, which allows them to withstand exposure to a variety of chemicals and solvents. This property is important in applications where the fibers are exposed to harsh environments, such as in the chemical industry.

Thus, the higher strength, consistency, purity and chemical resistance of cellulose fibers make them a desirable choice over many natural fibers for a wide range of applications.

Table 1. Mechanical and structural proprieties of sisal fibers and cellulose fibers.

	diameter range (μm)	diameter average (μm)	Tensile Strength σ (MPa)	Increase of σ (%)	Young's modulus E (GPa)	Increase of (E) (%)
Sisal fibers	80-220	125	125	--	62	--
Cellulose	40-75	57	530	324	131	111

3.2 Chemical analysis

The use of EDX spectral analysis is a powerful method for identifying the elemental composition of materials, especially in complex samples such as biological tissues, geological materials and synthetic polymers. This technique can help determine the chemical and physical properties of materials and assess their suitability for various applications. In this research, EDX analysis was performed on natural sisal fibers and cellulose, two types of sisal fibers with unique properties such as biodegradability, renewability, and mechanical strength which make them attractive for various applications such as composites, textiles, and packaging materials.

The results shown in Fig. 1, indicate that the two samples have different compositions. The natural sisal fibers contain a higher percentage of carbon, oxygen, and calcium compared to cellulose. Percentage by mass is the amount of a given element in the total mass of a material. Specifically, natural sisal fibers contain 35.14% carbon, 47.09% oxygen, and 1.34% calcium, while cellulose contains 36.79% carbon, 29.22% oxygen, and 0.11% calcium. This indicates that cellulose fibers have a much lower amount of calcium than natural sisal fibers. These results are significant because they provide insight into the elemental composition of these two materials, which may have implications for their properties and potential applications.

The higher carbon content in cellulose indicates that the treatment has increased the degree of carbonization, possibly by removing non-cellulosic materials such as lignin and hemicellulose. The higher nitrogen content in cellulose may be due to the incorporation of amino acids, which can react with alkali during the extraction process. The presence of calcium suggests that the extraction process may have caused a chemical reaction with the calcium ions in the treatment solution, resulting in the incorporation of calcium into the fiber structure.

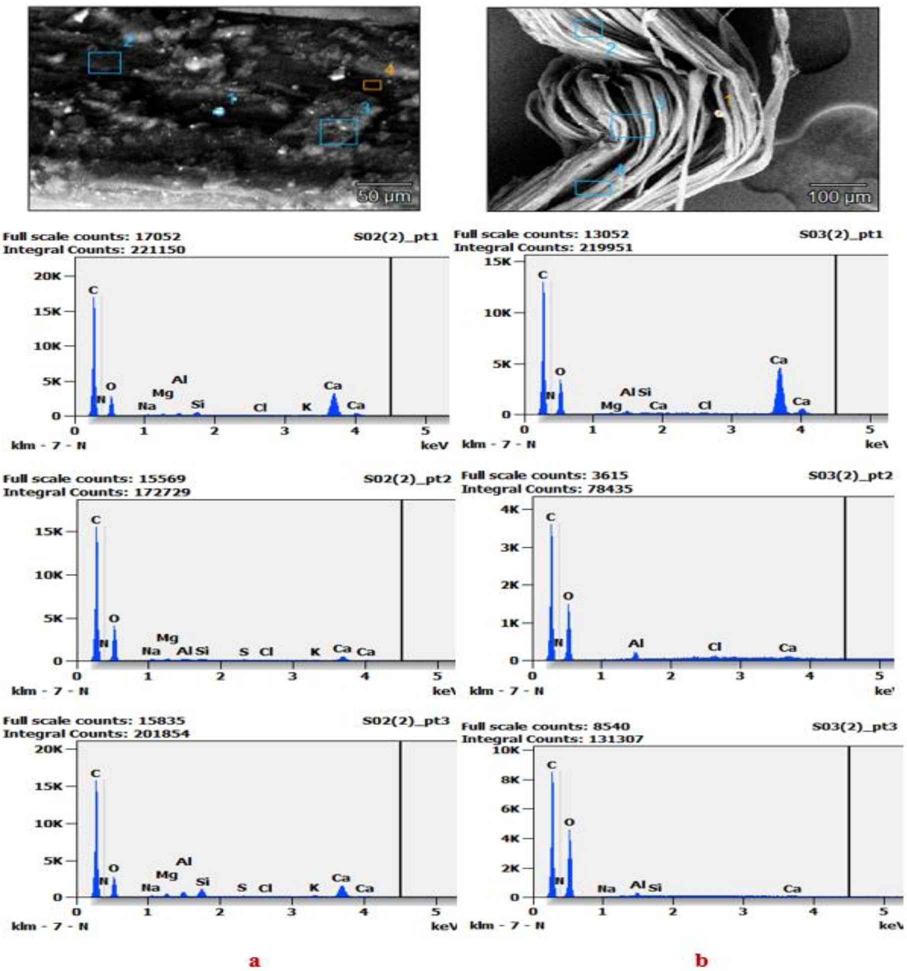
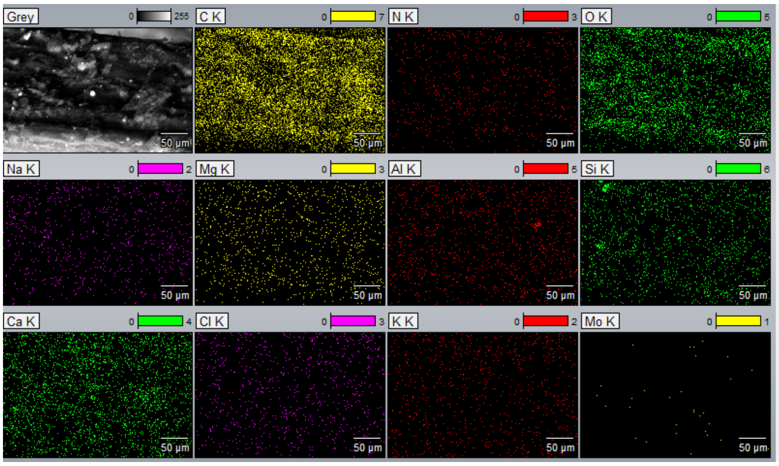


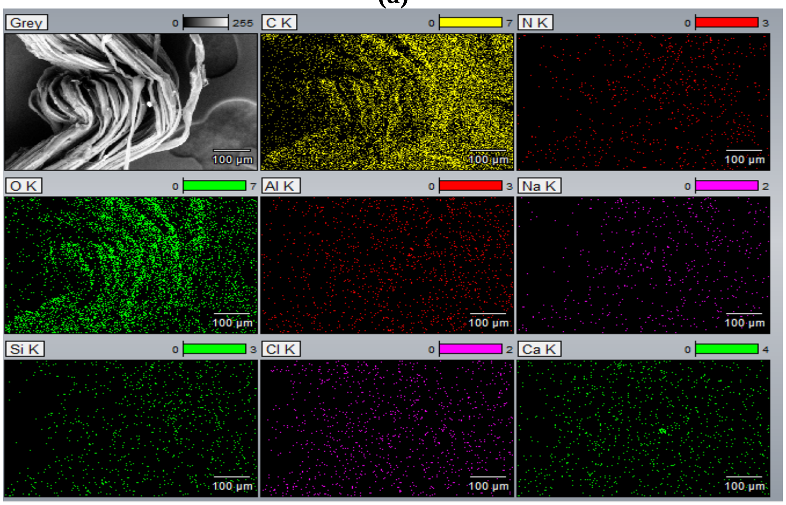
Fig. 1. EDX analysis for (a) natural sisal fibers and (b) cellulose extracted from sisal fibers.

The different chemical compositions of the two samples can have significant effects on their properties and potential uses. For example, the cellulose fibers have a higher amount of carbon, which may make them more suitable for high strength applications such as composites, while the natural sisal fibers have a higher concentration of calcium, which may make them more suitable for agricultural applications such as animal feed or soil modification. Fig. 2 shows the differences in elemental composition between the two samples, with the cellulose fibers occupying a distinct area of the graph from the natural fibers.

By using EDX for spectral analysis, valuable insight can be gained into the elemental composition of cactus fibers and a comparison can be made between natural and cellulosic fibers. This information can help in understanding the properties of these fibers and their potential applications in various fields. The differences in chemical composition between the two samples suggest that the alkali treatment has significantly altered the structure and properties of the fiber, which could have important implications for its potential applications.



(a)



(b)

Fig. 2. Elemental mapping of EDX analysis for (a) natural sisal fibers and (b) cellulose extracted.

3.3 Thermal analysis

3.3.1 TGA

TGA (Thermogravimetric Analysis) is a commonly used technique to study the thermal properties of materials. In this case, we analyzed two types of sisal fibers: natural fibers and cellulose extracted from these fibers.

Fig. 3 shows the results. For the natural fibers, part 1 of the curve shows a small weight perturbation, probably due to the evaporation of adsorbed water. Part 2 is characterized by mass stability up to a temperature of about 260°C, which can be explained by the presence of lignin and hemicellulose in the fibers. Part 3 shows a significant mass loss of 34% up to a temperature of about 360°C, indicating thermal degradation of these components. Part 4 shows a small mass loss up to a loss of 45% at a temperature of 605°C before the mass becomes stable at this temperature. This mass loss can be explained by the degradation of cellulose, which is the main component of sisal fibers.

In contrast, for cellulose extracted from sisal fibers, part 1 of the curve also shows a small weight perturbation. Part 2 is characterized by mass stability up to a temperature of about 300°C, probably due to the high purity of the extracted cellulose. Part 3 shows a significant mass loss of 26.5% up to a temperature of about 482°C, indicating thermal degradation of the cellulose itself. Part 4 shows a small mass loss up to a loss of 31.5% at a temperature of 615°C before the mass becomes stable at this temperature which confirms the findings of M. K. Haider et al [21].

In summary, this comparative TGA analysis shows that cellulose extracted from sisal fibers has higher thermal stability than natural fibers, probably due to its high purity. However, both types of fibers undergo significant thermal degradation at high temperatures, mainly due to cellulose degradation. This analysis may have important implications for the use of these materials in various industrial applications, particularly as reinforcements in composite materials and papermaking.

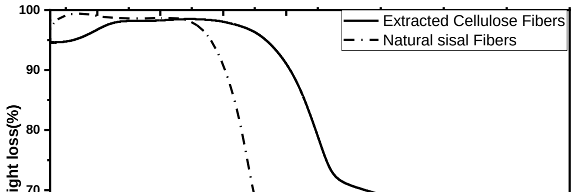


Fig. 3. TGA analysis for natural sisal fibers and cellulose extracted from sisal fibers.

3.3.2 DSC

The thermal properties of natural fibers and cellulose extracts have been studied using differential scanning calorimetry (DSC). DSC measures the heat absorbed or released during phase transitions such as melting or crystallization of the samples. In this study, DSC was used to investigate the thermal decomposition of fibers, as shown in Fig. 4.

The results show that natural fibers exhibited a 4% mass increase at 100°C due to evaporation of water present in the fibers. This was followed by a 7.7% mass loss at 140°C due to the decomposition of the organic components of the fibers. Mass loss continued up to 395°C, where mass loss increased to 53.3% due to the combustion of residual carbon after the decomposition of the organic components. The mass stability at high temperatures was 54.2% lower than the initial mass at 500°C.

On the other hand, cellulose showed an increase in mass of 2% at 90°C, followed by a steady decrease of 45.7% up to a temperature of 375°C before becoming stable at the temperature 550°C. These results are consistent and support the findings of Mannai et al [22].

The advantage of cellulose is particularly important in industries such as automotive and aerospace, where materials are exposed to high temperatures and must maintain their structural integrity. For example, cellulose-based composites can be used in high-temperature applications such as aircraft parts, brake pads, and engine components.

In addition, the thermal stability of cellulose makes it an attractive option for several other applications where heat resistance is important, such as in the production of fire-retardant materials, insulation materials and electronic components.

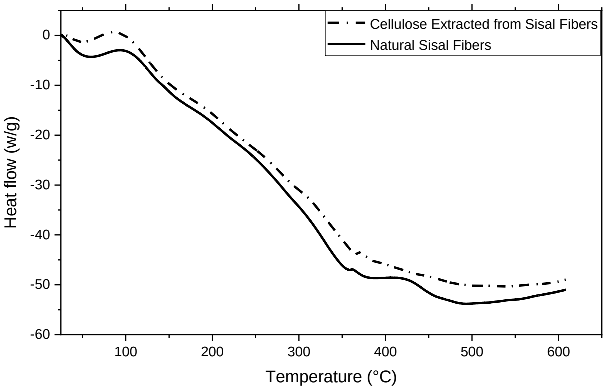


Fig. 4 DSC analysis for natural sisal fibers and cellulose extracted from sisal fibres.

3.4 Physical proprieties

The absorption rate of a material refers to its ability to absorb liquid or moisture. As shown in Fig.5, Tunisian sisal fibers have a much higher absorption rate than cellulose, as they can absorb up to 320% of their weight in water. This property makes it extremely useful in applications where absorption is essential, such as in the manufacture of absorbent materials like diapers or in erosion control mats. The structure of the fibers allows them to hold a large amount of liquid without breaking down or losing strength. In contrast, cellulose has a much lower absorption rate and can only absorb up to 18% of its weight in water. While this property may not be desirable in applications where high absorption rates are required, cellulose has other valuable properties. For example, cellulose has high tensile strength, which makes it useful in the manufacture of paper products, textiles, and medical implants.

Density is a measure of the mass of a material per unit volume. Sisal fibers have a lower density of 1.41 compared to cellulose, which has a density of 1.56. This means that sisal fibers are lighter than cellulose, which can be an advantage in applications where weight is a critical factor. However, the lower density of Tunisian sisal can also make it less durable than cellulose, which has a higher density.

The unique properties of Tunisian sisal and cellulose fibers make them suitable for a wide variety of applications. Tunisian sisal fibers are ideal for applications where absorption is critical, such as in the manufacture of absorbent materials like diapers and in erosion control mats.

Cellulose, on the other hand, is ideal for applications where tensile strength is critical, such as in the manufacture of paper products, textiles and medical implants.

4. Conclusion

In conclusion, the extraction of cellulose from sisal fibers has the potential to create sustainable materials that can be used in various industrial applications. This study showed that the extracted cellulose has good mechanical properties and thermal stability, making it suitable for use in high temperature and harsh chemical environments. In addition, the use of natural fiber waste as an alternative to synthetic materials can reduce environmental impact and promote circular economy practices.

The benefits of cellulose in natural fibers, such as biodegradability, renewability, light weight, and chemical resistance, make it a versatile and sustainable material that can be used in a wide range of applications. This study contributes to the growing body of research on sustainable materials and highlights the potential of using natural fiber wastes in industry, including the potential use of sisal fibers in other areas such as biodegradable packaging materials, textiles, and medical applications.

The development of sustainable materials is crucial to addressing environmental challenges and promoting sustainable development, and this study provides valuable insights into the use of natural fibers as an environmentally friendly alternative to synthetic materials. Further research in this area can lead to more sustainable materials and a better future for the environment.

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