Extraction and physical characterization of a novel cellulosic fiber derived from the bark of daphne Gnidium plant

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Research Article

Keywords: plant fiber, composite materials, physical properties, fiber extracting, daphne gnidium

Posted Date: February 20th, 2024

DOI: https://doi.org/10.21203/rs.3.rs-3884803/v1

Additional Declarations: No competing interests reported.

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Received: date / Accepted: date

Abstract The primary aim of this research is to develop a method for extracting fibers from the bark of the Daphne Gnidium shrub for use in various fields, such as paper manufacturing and reinforcing composite materials with plant fibers. These plant fibers have gained significant attention and are increasingly utilized in automotive, infrastructure, and renewable material-based sectors. Following the extraction of Daphne Gnidium bark fibers using the method outlined in this study, we analyzed their morphological, chemical, and physical properties in samples of the biofibers, locally known as Lazaz. The characteristics (physical, chemical, and morphological structure) revealed in this research align with numerous other studies, particularly in terms of density. We also consider the economic and environmental contexts of local natural products in Algeria, prompting us to undertake comprehensive studies in the environmental field to identify new types of useful plants that can contribute to technological development.

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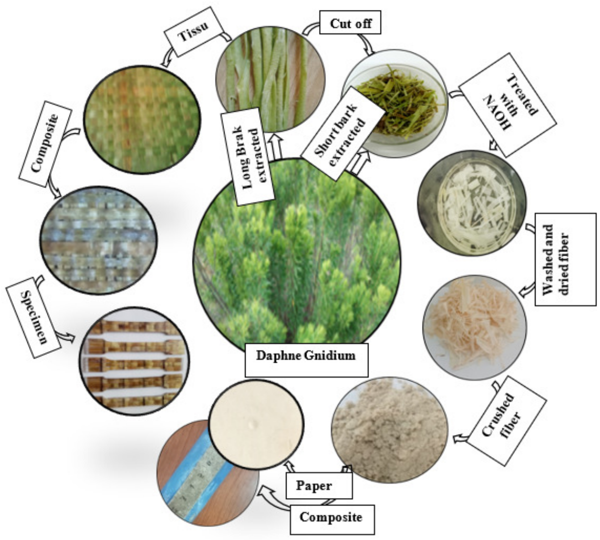
Graphic abstract



Keywords plant fiber · composite materials · physical properties · fiber extracting · daphne gnidium

1 Introduction

The future generation's sustainable tomorrow depends on current industrial development and environmental efficiency. These factors are reflected in the quality of products and their ability to meet needs and requirements, including strength, durability, light weight, ease of manufacturing processes, biodegradability, and other advantages. Natural fiber-based composite materials such as sisal, jute, Abaca, coir, hemp, etc., have enabled many innovations by improving physical and mechanical properties such as density [1], elastic modulus [1, 2], strength at the elastic limit and/or at break [3], etc. The family of fiber reinforcements of composite materials, which appeared in the forties of the last century, continued to develop due to their mechanical properties that are often associated with low density[4]. In recent years, the use of composite materials made from natural fibers has spread so quickly that they have become ubiquitous in our daily lives through their application in various fields such as the automotive industry [5,6], furniture and construction[7], electrical and nuclear industries[8], aviation [9,10], transportation [11,12], and medical fields [13]. Scientific research in recent years has focused on nature-inspired alternative solutions. Although the use of plant fibers is not new, according to M. Supran Salit, Egyptians have been using natural fiber compounds for about 3,000 years [14]. Therefore, renewable plant materials can form a new platform for sustainable, industrially advanced, technologically, and environmentally efficient progress. Fiber is a filamentous material that is much longer than it is wide, with very small diameters [15]. It varies in length and is used to reinforce polymeric compounds. It is classified as a natural fiber [16]. Natural fibers are usually characterized by certain properties; they are biologically dissociated and can be obtained from bio-renewable sources. Synthetic fibers, on the other hand, are characterized by their strength, durability, low cost, and greater possibility of modifying their properties and chemical composition. The structure of natural and synthetic fibers is completely different. In addition, there is another category that falls between the two (natural and synthetic) and shares certain characteristics for both categories named renewable fibers, which includes cellulose fibers [17]. Natural fibers are classified into three varieties: plant fiber, animal fiber, and mineral fiber[18].However, plant fibers are divided into six categories, grain, leaf, fruits, bark, root and stem fibers (Fig. 1).

1.1 biofiber components and its displacement

Plant fibers are fibrous biological structures of plant origin[19], consisting mainly of cellulose, hemicellulose and lignin[20, 21] in relatively lower proportions than extracted materials, proteins and some inorganic compounds[22] (Fig. 2). The proportions of these different components depend largely on the species, age, and organs of the plant[19, 20]. There are several factors that affect the morphology of plant fibers, depending on the origin of biomass, the environmental conditions of plant growth, and the state of maturity of the latter. Plant fibers also vary according to the diversity of their cell wall thickness. Plant fibers are widely used in industrial fields due to their high physical and mechanical properties, being eco-friendly[23], low-cost[24] and lightweight[25] and their chemical composition (cellulose, hemicellulose and lignin) playing a major role in determining their properties and characteristics [26].

Biomass refers to chemical elements derived from natural sources, and is composed of substances such as lignocellulosic materials. These materials contain cellulose $(40-60\%)$, hemicellulose $(20-35\%)$, and lignin (10-25%) [28, 29], and are an integral part of plant cell walls. Due to their complex structure, they are utilized in various industries such as paper, textile, and polymer production [30]. Statistics research suggests that approximately 950 million tons of biomass are produced annually in Europe [31].

1.2 Structure of plant fibers

The cell wall of plant fibers consists of several main layers, namely the middle lamina, the primary wall, and the secondary wall, as shown in Fig. 3 [32], where the primary wall is the first wall formed during cell growth and the middle lamina works by connecting the cells to each other, while the secondary

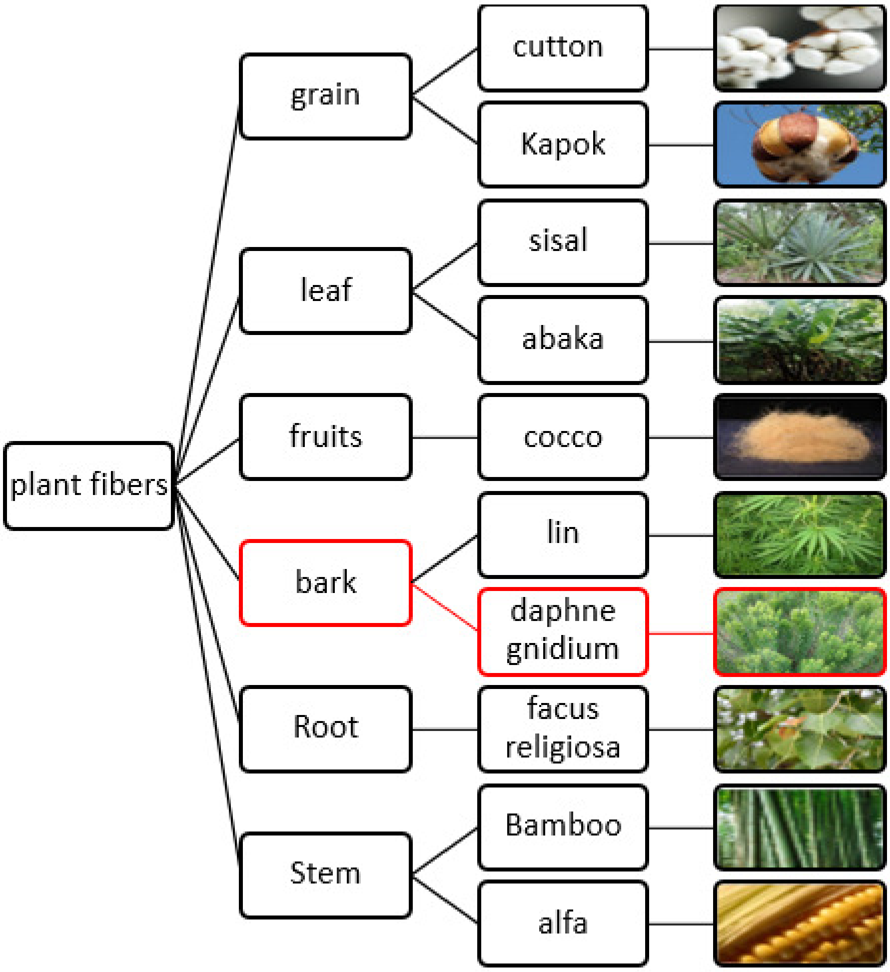


Fig. 1 Diagram depicting fiber classification

wall is divided into three sub-layers that differ from each other naturally and chemically (S1, S2, and S3)[29,33].

2 Materials and method

In this work, we used sodium hydroxide solution, which is a strong chemical base with the chemical formula $NaOH$, also known as caustic soda, and is used in many industries. Its solubility and ability to completely dissociate in water are very high, and it is characterized by many properties, including a molar mass of 39.9971 g/mol, a volume mass of 2.13 g/ml, a melting point of 318 °C,

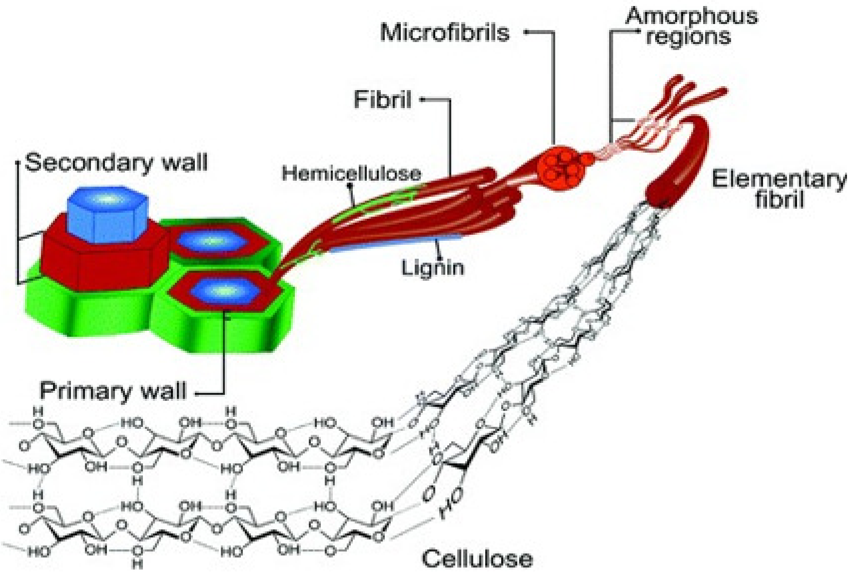


Fig. 2 Structure of plant fibers [27]

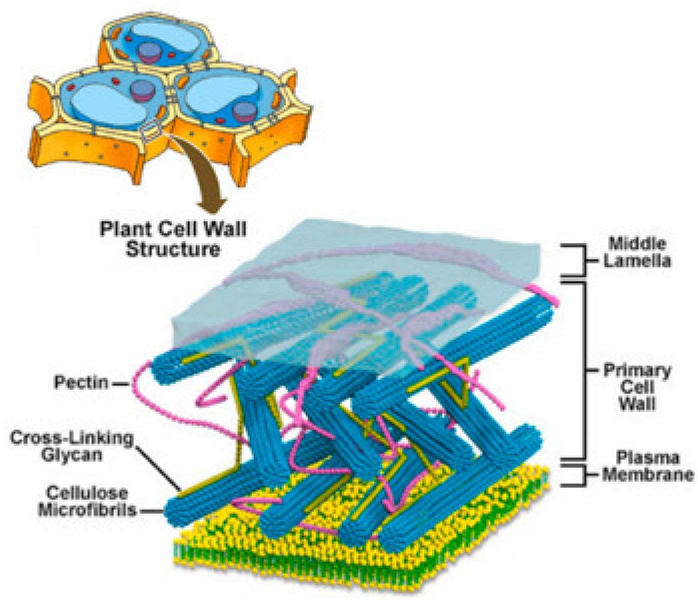


Fig. 3 Plant cell wall forming layers [32]

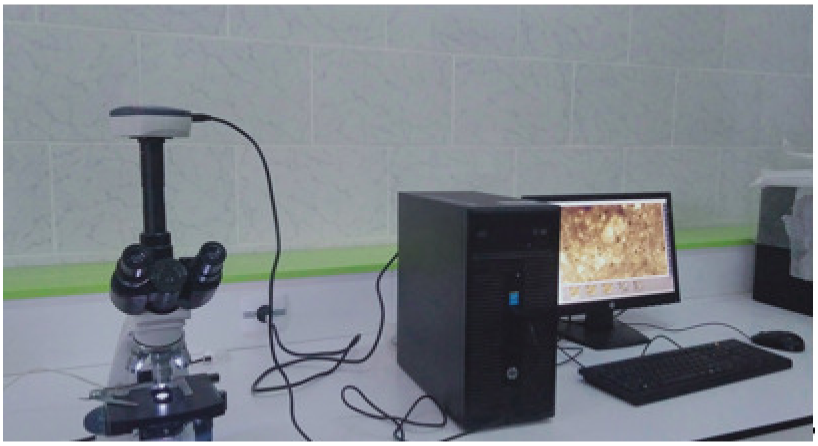


Fig. 4 Optical microscope assisted with computer

and a boiling point of 1388 °C. We also used distilled water and the bark of the Daphne Gnidium plant. To prepare the bio-fibers, we used the following devices:

2.1 Optical microscope

It is a type of microscope that uses visible light and a lens system to magnify images from small samples. The specimen to be examined is placed on the stage platform under one of the body lenses. The perforated platform is illuminated from its center with a concentrated light source with an intensive lens. A mirror and an external lamp or daylight are used to illuminate the sample placed on the platform. This light microscope is equipped with a camera that is connected to a computer and is accessible at the level of the science laboratory at the Higher School of Teachers of Technological Education (Fig. 4).

2.2 Infrared device (SHIMADZU-FTIR 8400S)

In the Laboratory of Organic Synthesis and Biocatalysis (LSBO) of the Department of Chemistry of Badji Mokhtar Annaba University, we used the SHIMADZU-FTIR 8400S infrared spectrophotometer (Fig. 5) to analyze specimens of pellets prepared (2-4mg) in 200–225 mg of KBr. Infrared spectroscopy, or infrared spectroscopy, is a technique that is still widely used and is one of the branches of spectra that deals with an infrared region. It includes a set of techniques, the most famous of which is absorption spectroscopy. It can also be used to report changes within the crystal and is also used to identify functional aggregations that enable the manifestation of structure and chemical bonds.

Fig. 5 SHIMADZU-FTIR 8400S infrared spectrophotometer

Fig. 6 UN30 electric oven

2.3 Convection oven

The purpose samples from fluids for study were dried using an UN30 electric oven (Fig. 6) with a maximum temperature of 300 C, located in the Physics Laboratory for Material Sciences at the Higher School of Teachers of Technological Education.

2.4 Melting point SMP30

The melting point SMP30 (Fig. 7) is a device that is specifically designed to measure and record the melting point temperatures of samples, which are placed inside small-sized tubes where three samples can be placed simultaneously. It has a maximum temperature of 400 degrees Celsius, it contains a digital LCD screen on which the value appears. This device (melting point

Fig. 7 Apparatus of melting point SMP30

Table 1 Apparatus, tools and materials used

Apparatus and tools	Materials
Electronic Scale	Sodium hydroxide NaOH
Electrical oven	Distilled water
Becher 1000ml	The bark of the daphne
Electric heater	gnidium plant
Electric mixer	Potassium bromide KBr
Optical microscope	
Included Pipette	
Melting point SMP30	

SMP30) is housed in the Physics Laboratory for Material Sciences at the Normal High School of Technological Education. The table 1 represent the devices and materials used in physical experiments

3 Preparing the raw material

The sample was collected from the Daphne gnidium L plant in the Es-sebt area of Skikda state (Fig. 8), where this type of plant is plentiful and naturally distributed in northeastern Algeria, particularly in Skikda Province.

The plant (Fig. 9a) is harvested at the end of April and the beginning of May to aid in separating the bark with a knife (Fig. 9b). The bark extraction process should occur within 24 hours of harvesting. After harvesting, the bark

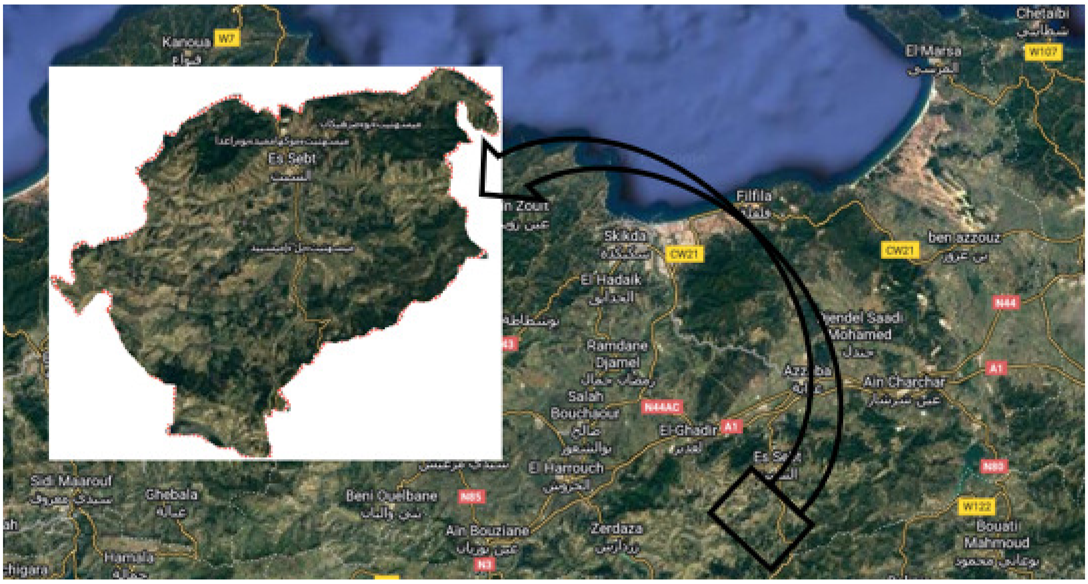


Fig. 8 Harvesting area of Daphne Gnidium

is removed from the plant's branches, and the outer peel is separated from the bark (Fig. 9c). It is then washed with distilled water to remove any remaining peel residue, and left to air dry for 12 hours. Subsequently, it is placed in a laboratory thermal dryer oven for 4 hours at 60 degrees Celsius until fully dried. Once dried, the bark sample is ready for use (Fig. 9d).

4 Extraction of Daphne Gnidium fibers

Daphne Gnidium bark fibers underwent a three-stage extraction process: chemical, washing, and mechanical. In the chemical stage, a 25% concentration solution of sodium hydroxide (NaOH) diluted with distilled water was used to modify the cellulose molecular structure, as depicted in chemical equation 1. This treatment gradually eliminates hemicellulose, lignin, pectin, and various oils from the bark, resulting in clean fiber surfaces. The color of the bark changes to white, while the fiber diameter decreases and their length increases. This enhances the fiber surface's effectiveness in adhering to the material to be reinforced.

$$liber + NAOH \rightarrow fibre + H_2O + Na^+OH^- + hemicellulose + lignin + pectin \tag{1}$$

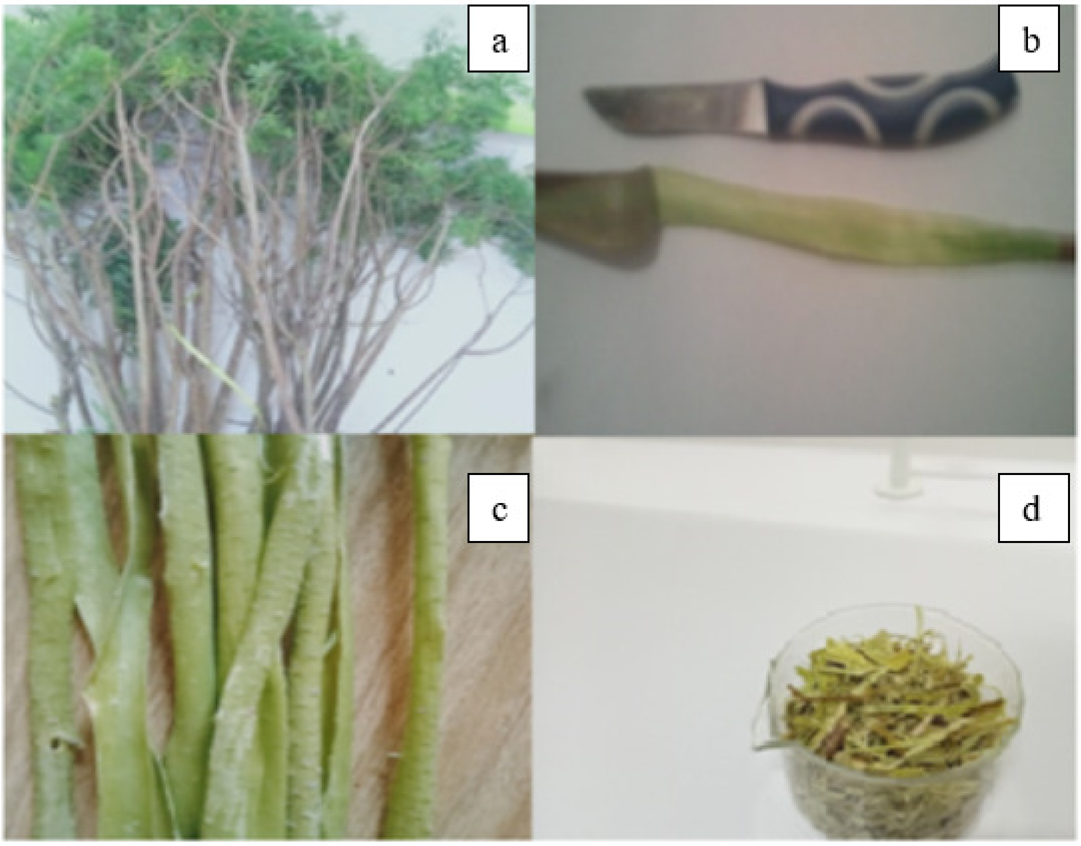


Fig. 9 Sample preparation steps

4.1 Chemical treatment

Chemical treatment is the most common method, in which the fibers are treated with sodium hydroxide (NaOH). In this process, we followed these steps: In a 1000 ml beaker, 200 ml of distilled water is added, followed by the addition of 100 ml of sodium hydroxide (NaOH) solution using a clean graduated pipette. Then, 40 g of chopped bark is added to the mixture, and the beaker is placed over an electric heater at a temperature of 80°C. Stirring with an electric mixer is done to accelerate the separation of cellulose fibers from other components (Fig. 10).

4.2 Washing stage

In order to prepare the fibers separated from each other, we use the grinding method, which is the mechanical stage. We take the sample that was previ-

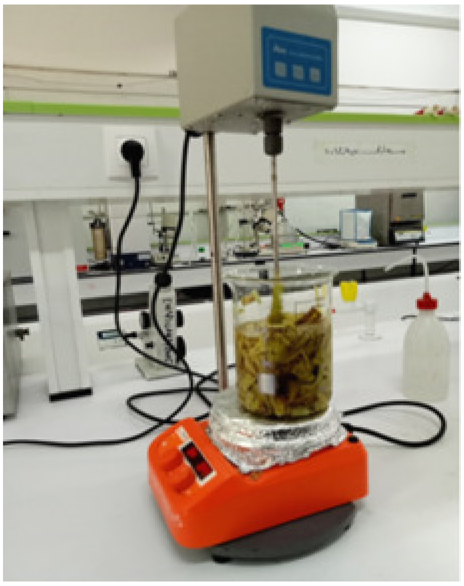


Fig. 10 Photograph showing the separation of cellulose fibers from other components of the bark

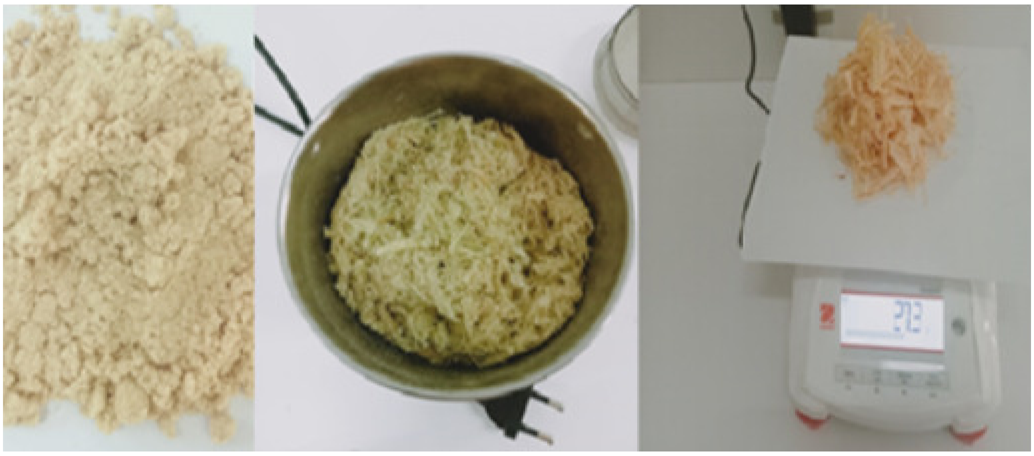


Fig. 11 Grinding and extracting fibers

ously treated, wash it with distilled water, dry it in a convection oven at a temperature of 60 °C for 4 hours, and grind it using a grinding device as shown in (Fig. 11).

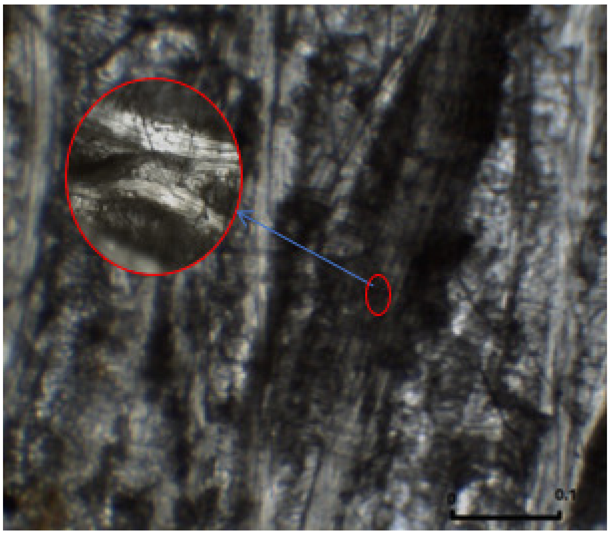


Fig. 12 Optical microscope image of cellulose fibers of Daphne Gnidium L

5 Results and discussion

The results and discussion of this work are about the morphological structures of bark fibers under an optical microscope and their physical properties, which are presented in the melting point and density of extracted fibers. Finally, we studied and discussed chemical properties using infrared spectroscopy

5.1 Fiber's morphological

Fig. 12 depicts the morphological structure of cellulose fibers under an optical microscope before the mechanical stage at a magnification of 10 μm per 1 mm (1 mm per 1028 pixels), where their diameters ranged from 10 to 25 μm.

5.2 Melting point of extracted fibers

To determine the suitability of using fibers extracted from the bark of the Daphne Gnidum plant in specific fields or under particular thermal conditions, it is essential to conduct tests on its melting point. This property holds significant importance, particularly for plant fibers, in the realms of physics and material science. At the High School for Teachers of Technological Education, we conducted an experiment using the Melting Point SMP30 device to measure the melting temperature. A sample of the material was placed inside a glass tube with a diameter of 1 mm and a length of 50 mm, which was then inserted into a designated hole. Upon completion of the experiment, the determined melting point was 297°C.

Table 2 Comparison of the density of some plant fibers

Plant fibers	flax	kenaf	jute	Abaca	Sisal	Coir	Cotton	Daphne Gnidium
density (gr/cm3)	1.42–1.52 [35]	1,4 [36]	1.5 [37]	0.75-0.995 [38]	1,45 [39]	1,15 [40]	1,5 [41]	0.577 [work]

5.3 Density of extracted fibers

We employed the Pycnometer technique to determine the density of fibers obtained from the bark of the Daphne Gnidium at a temperature of 24°C. To conduct this measurement, we require a flask of known volume, a non-polar solution with a known density (acetone), and a precise electronic balance. Density measurement of fibers went through four steps (Fig. 13); Initially, we weigh the empty flask and then pour the acetone into the flask (Pycnometer) as a non-polar solvent, with a its density is $\rho_a ce = 0.784 g/ml$. Secondly, subsequently, we place the flask filled with acetone on the electronic balance to ascertain the mass of the acetone along with the flask. Thirdly, we then measure the weight of a quantity of fibers. Lastly, we weigh these three components (fibers, acetone, and flask) using a high-precision electronic balance. The relative density of the fibers (D_r) obtained by executed of equation 2, and employing equation 3 for derive the volumetric mass of the fibers (ρ_f).

$$D_r = \frac{M_3 - M_1}{M_2 + M_3 - M_4 - M_1} \tag{2}$$

$$D_r = \frac{\rho_f}{\rho_{ace}} \Rightarrow \rho_f = \rho_{ace} * D_r \tag{3}$$

where: ρ_f: fibers density (gr/ml)
ρ_{ace}: acetone density (gr/ml)
D_r : relative density
M_1 : the mass of empty flask (gr)
M_2 : the mass of flask filling with acetone (gr)
M_3 : mass of fibers (gr)
M_4 : mass of flask filling with acetone and fibers (gr)

The density of the bark fiber of the daphne gnidium plant is equal to 0.577 g/ml as determined by the previously applied methods and rates. This result is comparing and consensus with the information found in specialized references (Table 2). we can conclude that the density of this bark fibers has a very important value. For example, the volumetric mass of the fibers of the artichoke is equal to 1,000 g/ml [34], by comparison, we can save approximately 50% of the weight.

5.4 Study of chemical properties by infrared spectroscopy

The aim of this study was to determine the functional groups of the organic chemical composition of fibers extracted from the bark of Daphne Gnidium

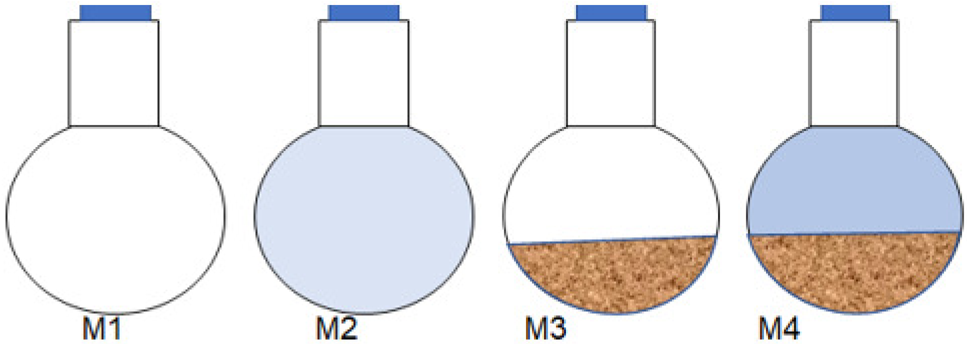


Fig. 13 Measurement of density by the Pycnometer method

plants. A sample of fibers extracted from the bark of the Daphne Gnidium plant is shown in an infrared spectrometer, in which pellets (2–4 mg) are prepared from the crushed sample with the addition of (200–225 mg) of KBr. In this study, a SHIMADZU-FTIR 8400S infrared spectrophotometer was connected to a computer. Spectral peaks were analyzed and interpreted using LabCognition's irAnalyze program, which contains a database for functional group identification. In the Laboratory of Organic Synthesis and Biocatalysis (Laboratoire de Synthèse et Biocatalyse Organique LSBO) of the Department of Chemistry at Badji Mokhtar Annaba University, a curve (fig 14) representing the instantaneous transformation (de Fourier transform) of the infrared spectrum was obtained.

The OH stretching vibration of cellulose and hemicellulose was observed at a wavelength of 3400 cm-1. The absorption peak at 2920 cm1 represents the CH vibration. On the right is also a peak whose wavelength is 1427 cm1 for the CH2 group vibrations. On the left is also a stretching of the C–O vibrations at the peak 1103 cm1, which represents the hydroxide compound group.

The sulfoxy atom vibration range in lignin extends to 1057 cm-1. For the chemical classification of the side chain, the absorption peak 1427 cm1 represents the bending of CH2 in lignin and the peak 2920 cm1 represents the O–H stretching in the functional group of aliphatic ether or sulfoxide (fig 15).

The small peak at 667 cm1 represents the extension of C–S and the peak at 1032 cm1 represents the extension of S=O. These two peaks are formed with the two modes of deformation for CH, which appear in the peaks 1427 cm1 and 1920 cm1 for CH alkalyl, which represents the aliphatic sulfoxide functional group.

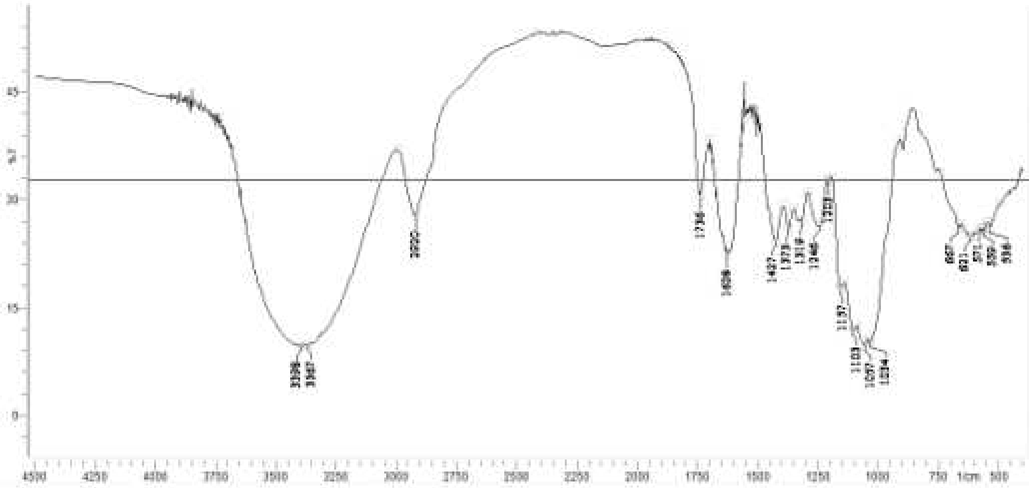


Fig. 14 Infrared spectroscopy of Daphne Gnidium fibers

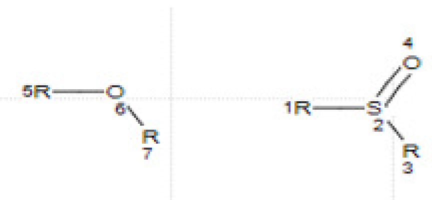


Fig. 15 Sulfoxide structure

6 Conlusion

In this study, we extracted the fibers that make up the bark of Daphne Gnidium through three stages: chemical, washing, and mechanical stages. The sodium hydroxide (NaOH) solution was diluted with distilled water in the chemical phase. Infrared spectroscopy was used to study its physical (density and melting point) and chemical properties. The study of plant fibers and their physical and chemical properties plays a major role in determining the type of fiber to be used to achieve desired results and intended purpose. In this study, the melting point of Daphne Gnidium fibers reached an important value, enabling them to be used at temperatures exceeding 200°C. In addition, we identified the functional groups by IR spectroscopy It makes it possible to know which materials can be used as reinforcement. On the other hand, the natural abundance of plants and the small density of their fibers compared to their counterparts encourage us to use them in several fields, including paper manufacturing, mechanical installations, clothing, etc.

Acknowledgements We gratefully acknowledge support from the Laboratory of Organic Synthesis and Biocatalysis (LSBO) under the supervision of Pr. khorief nassreddine abdelmalek for IR analysis. The authors also gratefully thank Pr. Toufik Benmansour for technical assistance.

Declarations

Conflict of interest The authors declare that they do not have any conflicts of interest.

Ethical Approval The research presented in this article does not involve any experiments involving human participants or animals conducted by the authors.

Consent for publication All authors consent for publication of the submitted manuscript.

Availability of data and materials Not applicable.

Funding Open access funding provided by Higher normal school of technological eduction Skikda ENSET.

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