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A STUDY OF PROPERTIES OF FIBERS EXTRACTED FROM KARKADE (HIBISCUS SABDARIFFA L.) AGRICULTURAL WASTE AS POTAINTIAL REINFORCING MATERIAL

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ABSTRACT

The use of agricultural waste in fiber production is increasing these days, due to its multiple benefits and ease of production. One of these agricultural wastes is the karkade plant stem. Sudan is considered as the country of origin of karkade plant, cultivated in a large area in north and west of Sudan to take its fruits and use in food and drinks and in some medical fields. After fruit picking, there is no use of the stem. This paper presents a research conducted to study the properties of karkade fiber as potential reinforcing material. For the purpose of the study, karkade fiber is extracted from karkade stem using water retting process with different duration times (15 to 30) days. Samples are prepared to be analyzed for chemical, physical, morphological and thermal properties. From obtained results it is found that karkade has good cellulose percent, acceptable moisture content and water absorption and density comparable with other natural fibers. Also, morphological analysis showed that it has a rough surface that supports bonding between fiber and matrix. From the above observation it can be concluded that karkade fiber properties make it promising for use as reinforcement in composite material.

KEYWORDS: karkade plant, agricultural waste, natural fiber properties, reinforcing material.

1. INTRODUCTION

Global attention to the environmental impact of industrial and agricultural waste has been increasing in recent years. The use of these wastes in industry has a dual effect of reducing the environmental impact of these wastes, and the production of sustainable alternative materials. Sustainable development has become a daily word these days [1] and the rareness of non-recyclable resources has attracted researchers pursuing to develop new eco-friendly materials and products based on sustainability principles [2]. Exploiting agricultural waste material will not only maximize the use of the agricultural products, but will also support preserve natural resources [3].

Natural fibers have been in consideration and attracted more attention in recent years [4]. Table (1) shows some types of natural fiber and country of origin. Natural fibers have many advantages like, low density, great specific strength and its renewability.

Natural fiber such as flax, kenaf, sisal, pine apple leaf and jute are now playing an important role in composite reinforcement [4]. Abaca fibers are used to replace glass fibers in the manufacturing of cars, planes and ships, and also used for building materials such as fiberboards, ceramic and tiles [6]. Devi, 1997 [7] reported that pine apple leaf fibers (PALFs) show excellent mechanical properties without any additional cost input. Also, kenaf fiber is a commonly used fiber which has many applications like paper products, building materials and absorbents [8]. On the other hand, for some other types of natural fibers, such as karkade, there is little information about their properties.

Table (1) Country of Origin of some Natural Fibers

No	Fiber	country of origin	References
1	Flax	Borneo	[5]
2	bamboo	Japan	[17]
3	Kenaf	Tanzania, Jamaica, Togo	[5]
4	Jute	India, Egypt, Ghana, Sudan	[5]
5	Sisal	India	[18]

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6	abaca	Indonesia	[19]
7	Karkade (Roselle)	Sudan, India, Malaysia, China	[14]

1.1 Natural fibers composites

In recent time many researchers conducted studies on natural fibers in general for their capability as reinforcement in composites. Fan and Fu, 2016 [9] stated that natural plant has high content of Cellulose which will produce good mechanical strength. Devi,1997 [7] examined the capability of using pine apple leaf fiber (PLAF) in composite material and reported that PLAF showed greater mechanical properties compared to other natural fiber. Also, from the experiment done on oil palm Kaliwon et al, 2010 [10] stated that oil palm EFB fibers have potential to use in a composite material in reinforced mortar roofing slates with the proper water cement ratio and concrete mix. Razali et al, 2015 [4] investigated the characterization of Roselle fibers at different ages and reported that Roselle fibers have comparable properties with other well-known fibers. Chauhan, 2013 [11] studied Roselle fiber properties and observed that the mechanical properties of Roselle fibers are improved when investigated using injection-moulded isotactic polypropylene/Roselle fiber composites. The study conducted by Hossen et al, 2018 [12] explained the difference between treated and untreated jute composite and they found that treated composites gave higher mechanical strength compared to the untreated one.

1.2 Karkade plant and Karkade fiber

karkade plant, is from Malvaceae family and is supposed to be native to Africa, cultivated in Sudan, India, China, Malaysia and Taiwan (Puro et al., 2014 [13]; Mohammed et al., 2012 [14]). karkade (Roselle) plant is an important annual crop plant grown in tropical and semi-tropical zones. Sudan is considered as the country of origin of karkade (Roselle), mainly in the Kordofan and Darfur zones. karkade (Roselle) may have been domesticated in western Sudan before 4000 BC [14]. karkade plant (Figure (1)) has many uses and its fruit is commonly used in the production of tea, jelly, jam, juice and medical applications. In Sudan Roselle, named karkade, is grown mainly by traditional farming methods in Kordofan and west Darfur. Sudan, Thailand and China are considered as major supplier of Roselle fruit in the world.

Figure (1) karkade plant and fibers, photo taken by corresponding author

2. MATERIALS AND METHODS

2.1 Materials

karkade plant had been harvested from traditional farms in North Kordofan state. karkade fiber was extracted using water retting process. After (15-30) days immersing in fresh water, stem was washed with clean water then fiber was removed manually, washed several times again and dried in sun light. karkade fiber was then prepared for testing.

2.2 Characterization Methods

2.2.1 Chemical Composition:

Chemical composition of natural fiber contains cellulose, hemi-cellulose, lignin and ash. The individual percentage of these components differs from fiber to another due to growing and harvesting conditions. Chemical

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composition of karkade fiber was determined using extraction method according to TAPPI standards (Tappi Test Methods 1992–1993).

2.2.2 Physical Characterization:
The physical properties of karkade fiber were obtained as follows:

Diameter:
Optical microscope was used to determine the diameter of karkade fiber, about ten samples, diameters were measured and the average value was obtained.

Density:
One of the most accurate methods to measure density is gas pycnometer method, 4.45g of powdered sample of karkade fiber was dried in an oven for 2h under 105oC, then analyzed 10 times using micrometers, at 24oC, under helium gas.

Moisture Content:
Moisture content is a significant property that affects classification of natural fiber. In this study moisture content determined by equation (i), following these steps:

First, 5 samples in powdered form were weighted as Mo. Next, weighted samples were heated in an oven for 24h under 105oC to eliminate the moisture. Then, the 5 samples were weighted again as M_1. Hence:

$$moisture\ content\ (\%) = \frac{M0 - M1}{M1} x100 \qquad (i)$$

Water Absorption:
Five samples of karkade fiber were prepared and weighted as M_o before immersing in fresh water over night in room temperature, then weighted as M_1. Water absorption was measured using equation (ii) in accordance with ASTM D570.

$$water\ absorption\ (\%) = \frac{M0 - M1}{M1} x100 \qquad (ii)$$

2.2.3 Morphology analysis (SEM)
Morphology analysis of karkade fiber was carried out using scanning electron microscope (SEM), to study the microstructure of fibers.

2.2.4 Thermal properties
To evaluate thermal stability and thermal decomposition of karkade fiber, thermo-gravametric analysis was carried out to know the compatibility of fibers with polymer through manufacturing (Figure (2)). Around 8 mg of karkade was analyzed under nitrogen N_2 atmosphere with temperature array from 50 to 600°C using heating rate of 10°C.

Figure (2) SDT Q600 V20.9 Build 20

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3. RESULTS AND DISCUSSION

3.1 Chemical Composition:

Mechanical properties of natural fibers are affected by cellulose content in fiber, as percentage of cellulose increases, stability and tensile strength of fiber increases. Table (2) shows the chemical composition of karkade fiber compared with different types of natural fiber. As shown karkade fiber contains a high percentage of cellulose (67.7%) compared to kenaf fiber (64.5%), and within range comparable to flax fiber (64-71.9%). On the other hand, the test reported low hemi-cellulose content (12.27%) compared to kenaf fiber (20.23%) and flax fiber (16.7-20.6%). These high cellulose content and low hemi-cellulose content are reflected on other karkade fiber properties.

Table (2) Comparison of Chemical composition of karkade fiber with that of other natural fibers

Fiber	cellulose	Hemi-cellulose	lignin	Ash	Resources
Karkade	67.7%	12.27%	12.21%	0.88%	Current study
Kenaf	64.5%	20.23%	6.21%	1.25%	[4]
Jute	60-62%	22-24	12-14%	-	[20]
Hemp	57-77%	14-22.4	3.7-13%	0.8%	[21]
Flax	64-71.9%	16.7-20.6%	2-2.2%	-	[22]

3.2 Physical Characterization

It is very difficult to detect a single karkade fiber with the naked eye, since a single fiber consists of a bundle of fibers, for that diameter of karkade fiber was measured using scanning electronic microscopeat 193× magnification with view field 715µm. The microscopic remark was achieved using a binocular Motic microscope with a camera and with driving software Motic Images2000. Diameter values varied between 110µm and 149µm, which is large compared to 50 to 80 µm reported by Nadlene, et al, 2015 [16].

The density of karkade fiber was measured using a helium gas pycnometeris to be 1.7 g/cm^3. The small amount of karkade fiber density reflects on specific properties of fiber, increasing specific tensile strength and modulus, also increasing specific flexural strength. This meets the requirements for using fiber in structural applications. Water absorption and moisture content of karkade fiber, shown in Table (3), are within range with other natural fiber.

Table (3) Physio-chemical properties of a selection of natural fibers

Fiber Type	Moisture Content%	Water Absorption%	Density g/cm^3	Sources
Karkade	1.26	70.43	1.56	Current Study
Kenaf	3.7	289	1.421	[4]
sisal	11	56	1.45	[23][25]
banana	-	60	1.35	[5][23]

3.3 Morphology Analysis (SEM)

Morphology of karkade fiber was carried out using scanning electron microscope mainly to identify the structure of a karkade fiber which consists of several elementary fibers, overlapped along the length of fibers and bonded together by pectin and other non-cellulosic compounds that provide strength to the whole bundle. Figures 3a, b, c, d show the entire structure of karkade fiber. Figure 3a displays the rough surface of karkade fiber which is appropriate for a respectable bond with polymer matrix. Figures 3 b, c and d are zooming of Figure 3a, showing that there are a lot of bur in the structure surface with impurities due to use of untreated fibers. Figures 3 (a) and (b) show also bundles of elementary fiber which is named technical fiber or single fiber, while the district at the interface of two cells is named mid lamella [4].

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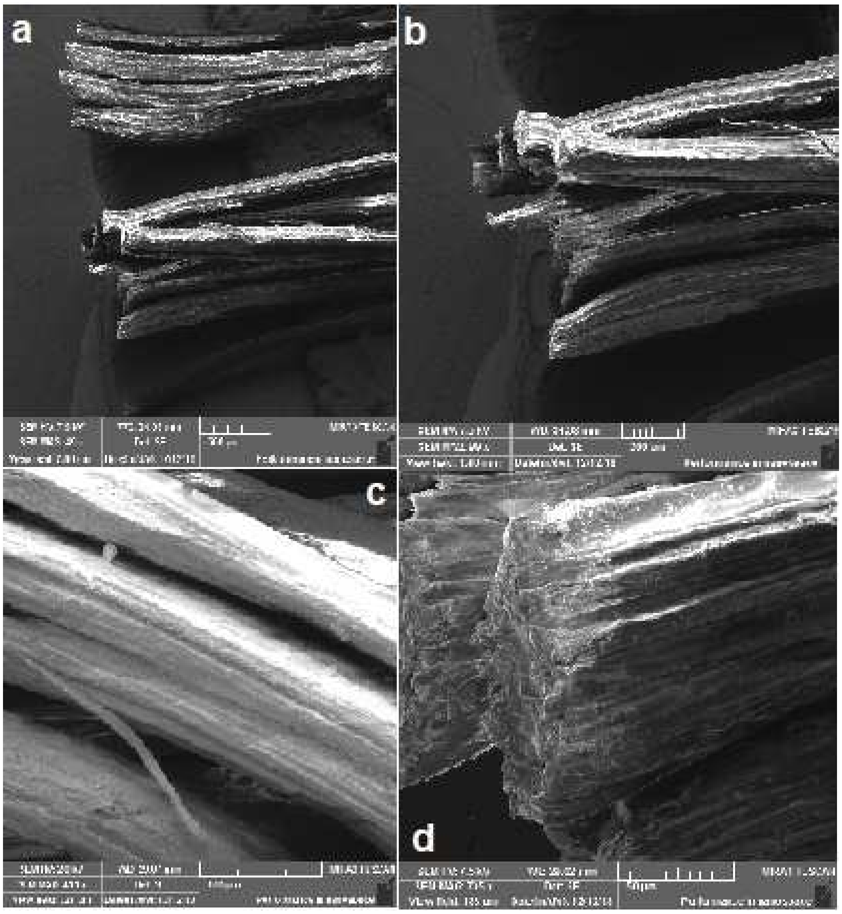

Figure (3) SEM micrographs of Karkade fibers bundle

3.4 Thermal properties

Thermoplastic natural fiber manufacturing methods need high temperature in their process. For that, the thermo-gravametric analysis was carried out to assess the thermal behavior of fiber before using in the fabrication of composites. In this study about 8mg of karkade fiber were tested to show their thermal stability. As reported by Belouadah, 2015 [15], natural fibers have two main phases of thermal degradation. The first one is a weight loss due to the water evaporation and the second phase is a decomposition of hemi-cellulose. The first phase recorded in this study occurred between 35^0-120^0C and as temperature increased, water evaporation occurred. It can be recorded that the decomposition of karkade fiber component happened between 220^0C-600^0C with two peak temperature points, in the first one at 350^0C the weight loss amount was 79.05% while the second peak temperature observed is 425^0C, (Figure (4)). As reported by Nadlene, et al., 2015 [16] the first component of fiber decomposed is hemi-cellulose followed by cellulose and finally lignin because of their toughness providing rigidity. It can be concluded from Table (4) that compared to other natural fibers, karkade fibers have good thermal properties that enable using it in composite fabrication.

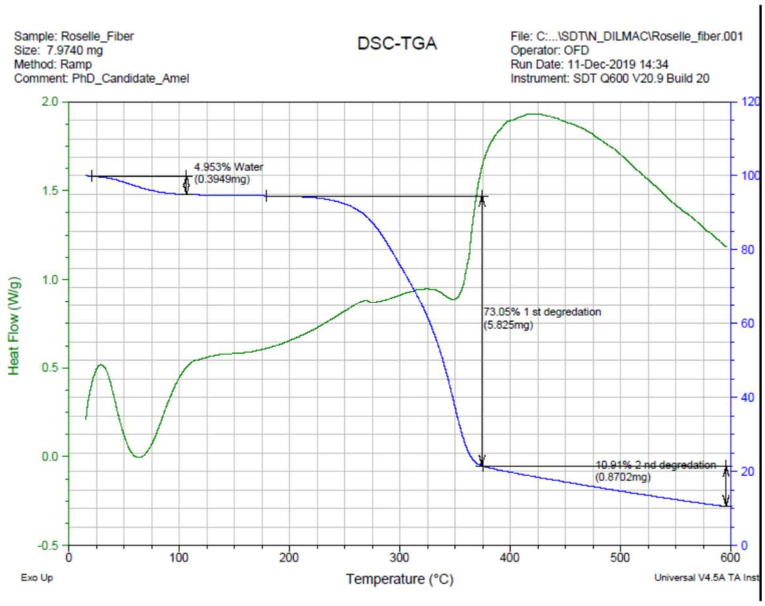

Figure (4) loss of weight and heat flow of karkade fiber

Table (4) Thermal Properties of Selected Natural Fibers

Fiber name	Initial degradation temperature^{0}C	Maximum degradation temperature^{0}C	Reference
karkade	220	425	Current study
Kenaf	219	284	[24]
Jute	205	283	[24]
Lygeumspartum L	220	338.7	[15]

4. CONCLUSION

This research was conducted on untreated karkade fiber to investigate the capability of using this fiber as reinforcement in fiber-reinforced polymer composite. The properties obtained included chemical composition, physical properties, thermal analysis, and morphology analysis. It was found that karkade fiber contains a high percentage of cellulose (67.7%), which leads to good stability of composite that reflects on mechanical strength. Moisture content and water absorption percentage of karkade fiber are respectable compared to most of the established natural fibers. The density recorded in this study is within range with kenaf, jute, and flax. Morphology analysis showed that the fiber has a rough surface with a bur. The degradation of karkade fiber started at 220^{0}C. From the above, it can be observed that karkade fiber properties are comparable with other recognized natural fiber and it can be used in the production of composite material. But there is still need to do a lot of research on treated and untreated karkade fiber to explore all its properties

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