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Original Research Article

Characterization of Honckenya (*Clappertonia ficifolia*) Fibre as a Potential Natural Fibre Reinforcement for Polymeric Composites

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ABSTRACT

Lignocellulose based natural fibres are gaining more acceptance because they are renewable, eco-friendly, cheap and possess good specific mechanical properties. These attributes have necessitated an increase in search for new vegetal natural fibres as reinforcement in polymer-based composites. Honckenya lignocellulose natural fibre was characterized to determine its physical, chemical, mechanical and thermal properties to establish its suitability in composites development. From the thermogravimetric analysis (TGA) investigations conducted, it was observed that the onset of thermal degradation was at 280 ^oC at 3% weight loss. The cellulose content was 63.48 ±1.1414wt% while lignin and hemicellulose contents were 7.91±0.12wt%, and 10.20±0.59wt% respectively. The density of the fibre was 1.22 g/cm^3, while the tensile failure strength was 677±36MPa, and the tensile modulus was 25.80±0.48 GPa. The Weibull distribution modulus, (m) was 2.66 and the characteristics strength, (σ_o) was 683.66 MPa. Furthermore, from the Weibull distribution, the Honckenya fibre has a reliability of 581 MPa at 50% survival probability. From the results obtained, it could be reasonably concluded that the Honckenya fibre is suitable as reinforcement in polymer- based composites development.

1. INTRODUCTION

The use of natural fibre as reinforcement for polymer- based composite is gaining wide acceptance in the industry especially in automotive sector (Jeyanthi and Rani, 2014). The wide acceptance and interest among researchers are based on the fact that natural fibres possess certain characteristics which are amenable to improvement as well as improvement of polymeric composites developed with such fibres (Chandramohan and Marimuthu, 2011; Faruk *et al.,* 2014).

It is interesting to note that natural fibres offer considerable advantages over synthetic fibres in certain areas of application which requires light weight and energy saving capabilities. For instance, natural fibres have good specific strength and specific modulus which are vital performance indicators in material selection processes (Faruk *et al.,* 2014). These major attributes have necessitated an increasing interest in research in the field of vegetable based natural fibre reinforced polymer composites (Pickering *et al.,* 2016).

Natural fibres of vegetable origin have proven to be the spotlight of emerging technology and development owing to the fact that it is a renewable and cheap source of natural resources (Pickering *et al.,* 2016). These potentials as highlighted have further led to an upsurge in the awareness of the inherent potentials of lignocellulose based natural fibres for composite applications. Just as the menu of conventional engineering materials keep on expanding because of the complexity and divergent demand of technical products and high-end materials to meet up with service requirements; the spectrum of natural fibre reinforced composites are also growing (Fiore *et al.,* 2014; Kumode *et al.,* 2017; Maache *et al.,* 2017; Vaisanen *et al.,* 2017; Manimaran *et al.,* 2018). Therefore, because of the wide range of potential areas of application for natural fibre reinforced polymer, - some of which have been established- new vegetable based natural fibres are continually being added to the menu of existing natural fibres (Chandramohan and Marimuthu, 2011).
In addition to the specific properties for which natural fibres offer great advantage in their usage in reinforcement of polymer matrix, lignocellulose natural fibres have good impact resistance properties (Jeyanthi and Rani, 2014). Research has shown that in crash circumstances, fenders and bumpers made of natural fibre reinforced polymer composites do not leave behind splinters which could further pose as hazard at the scene of the crash (Garkhail *et al.,* 2000; Mueller and Krobjilowski, 2004). In other words, natural fibres are considered for the development of crashworthiness components such as helmet because of the aforementioned reasons of non-splinter damage, as well as light weight of the helmet. Vegetable based natural fibre also have good sound proof characteristics, this has now made it possible for natural fibres to be used has polymer reinforcement in buildings and paneling for theatres and auditoriums (Mueller and Krobjilowski, 2004).

Natural fibres used in polymeric based composites are mostly of plant origin. However, other sources of natural fibres are from animals and mineral based fibres. Natural based fibres that have been used as reinforcement in polymer- based composites include Flax, Jute, Sisal, Hemp, Ramie, Coir, Henequen, Kenaf, Cotton, wool, chicken feather and Asbestos. There are vast arrays of natural fibre yielding plants mostly lignocellulosic, many of which have not been explored for composites reinforcement. This potential makes research on lignocellulose based natural fibre reinforced composites very attractive. Many researchers are continually exploring the possibility of new natural fibre usage in composite development and many of these research efforts have resulted in new composite materials development from such fibres (Al-Oqla and Sapuan, 2014; Binoj *et al.,* 2017; Maache *et al.,* 2017).

Vinayaka *et al.* (2017) studied the suitability of *Ricinus communis* (Castor plant) fibre as reinforcement for polymer composites. A study on characterization of *Furcraea foetida* new natural fibre as composites was conducted by Manimaran *et al.* (2018). Kumode *et al.* (2017) used balsa tree as potential reinforcement in preparation of green composites with castor seed cake. Binoj *et al.* (2017) used industrial discarded fruit fibre *Tamaridus indica L* as reinforcement in polymer composites. Ramasawmy *et al.* (2017) explored the use of chemically treated *Pandanus utilis* (screw pine) fibres for potential application as reinforcement in epoxy composite. Al-Oqla and Sapuan (2014) investigated the possibility of date palm fibre (DPF) and its sustainability in composite development for automotive industry; selection criteria for some of these natural fibres for composites development were also proposed by these researchers. Maache *et al.* (2017) characterized *Juncus effusus L* plant fibre of nothern Algeria origin in order to determine is morphological, physical, thermal and mechanical properties as well as its suitability for use in composites development. Honckenya fibres have been exploited in other areas of application such as fish net, ropes and mats. However, its use in composites development has not been exploited.

Honckenya with the botanical name *Clappertonia ficifolia* and commonly known as Mgbo in Igbo belongs to the plant family Tilaceae. The plant is very widely spread in continental tropical Africa and it is used for making mats, ropes, cordages and twine. It is a shrub that grows up to 3 m tall; the Flower is bisexual, while the sepal can grow up to 3.5 cm long and 6 mm wide. Clappertonia occurs from sea level up to 1200m altitudes in swamps, riverine and swampy forest, forest fringes and thickets. In fallow lands, it can become dominant or even form an almost pure, dense stand and these populations are often exploited for fibre production (Bosch, 2011; Brink and Achigan-Dako, 2012).

The objective of this research is to characterize Honckenya in order to establish its suitability to serve as reinforcement in polymer matrices. In order to investigate the potential of Honckenya fibre for polymer matrix composites, the following characterization were conducted: tensile strength, tensile modulus and tensile elongation, density, TGA, FTIR, chemical composition analysis after the fibre extraction.

2. MATERIALS AND METHODS

2.1. Material Collection

Honckenya fibre was obtained from Uburu, Ebonyi State south eastern Nigeria prior to planting season.

2.2. Fibre Extraction

The fibre extraction process involved two stage processes which were fibre separation or decortication and fibre retting process. The bark was separated from the Honckenya stem using sharp knife to manually peel off the bark. The Honckenya barks that were separated from the stem were retted in water for 30 days. The retted fibres were washed at the end of 30 days in order to obtain the fibre bundles. The retted fibres were sun-dried for a week and stored for further analysis. Plates I and II show the Honckenya bast fibre decorticated before retting and the fibre after retting (Mussig, 2010).

Plate I: Decorticated bast

Plate II: Honckenya fibre

2.3. Characterization of Honckenya Fibre

In order to establish the suitability of Honckenya to serve as reinforcement in polymer matrices to form composites, thermo-physical and chemical characterization of the fibre were conducted using thermogravimetric analysis (TGA), chemical composition analysis and Fourier Transform Infrared (FTIR) analysis. Compositional analysis of Honckenya was done using Fonnesbeck and Harris method for lignin, hemicellulose and acid insoluble (Van Soest, 1991), while the cellulose content was determined by Mynard method (Crampton and Maynard, 1938). Thermogravimetric analysis of Honckenya untreated fibre was conducted at FUT Minna, Nigeria, using a TGA 4000 Thermogravimetric Analyzer- Perkin Elmer apparatus at a heating rate of 10 ˚C/min under a nitrogen atmosphere, the temperature range of heating was from 28 ˚C to 887 ˚C. The weight loss as a result of the rise in temperature of the fibre was plotted on a thermogram. FTIR of Honckenya plant Fibres was determined using Agilent Cary 630 FTIR Spectrometer at Central Research Laboratory, chemistry department ABU, Zaria this is in order to establish the functional groups of the untreated Honckenya fibre.

2.4. Tensile Test and Weibull Distribution

The tensile test was conducted on fibre bundles according to ASTM D3822/D3822M-using AT-880B electric tensile testing apparatus with 500 N load capacity at Nigerian Institute of Transport technology, Zaria. The Honckenya fibre was attached to the jaws of the testing machine and the fibre manually aligned. The Honckenya fibre was of gauge length 40 mm, loaded to fracture at a testing speed of 1 mm/min with the fibre breaking force recorded. From the breaking force, tensile properties such as fracture stress, tensile modulus and fracture strain for the Honckenya fibre were evaluated. The fibre diameter was determined using a micrometer calibrated stage and ocular light microscope; the fibre diameters were recorded and subsequently used for the calculation of the fibre fracture stress. Tensile strength of natural fibres exhibits wide variation of values and brittle failure, hence the use of Weibull distribution to analyze the probability of failure of the natural fibres. The gauge length of the test sample was 40 mm with a sample size($n = 51$). The results of the tensile tests for Honckenya for the untreated fibres were analyzed for two parameter weibull. Equations 1 and 2 give the two parameter Weibull equation and the estimator respectively.

$$P_f(\sigma) = 1 - \exp\left[-\left(\frac{\sigma}{\sigma_o}\right)^m\right] \tag{1}$$

$$P_{fi} = \frac{i}{(n+1)} \tag{2}$$

where, $P_f(\sigma)$ is the probability of failure of the fibre, σ is the applied stress, σ_o is the characteristic strength or the scale parameter, m is the shape parameter which is also called the Weibull modulus, n is the sample size and i is the rank (Belaadi *et al.,* 2014; Fiore *et al.,* 2014; Maache *et al.,* 2017). The Weibull equation was transformed into linear equation which was plotted on the vertical axis against the failure stress.

2.5. Density Measurement

The density of the fibre was determined using Archimedes principles according to ASTM D792-13. Fibre samples were weighed in air with the weight recorded subsequently the fibre was then immersed in water. The weight of the measuring cylinder was first recorded, after which the weight of the cylinder and water was recorded, finally the weight of the cylinder plus water and the fibres were then recorded. Based on Archimedes principle, the immersed fibre was expected to displace a volume of water which was recorded. Equation 3 was used for the fibre density determination

$$\rho = \frac{m}{v} \tag{3}$$

Where, ρ is the density, m is the weight of the fibre and v is the volume displaced

3. RESULTS AND DISCUSSION

3.1. Honckenya Fibre Chemical Compositions

From the compositional analysis, the cellulose content of the Honckenya was 63.48±0.14wt%, while the hemicellulose content was 10.20±0.59wt% and the lignin content was 7.91±0.12wt%. Table 1 shows the compositional analysis of untreated Honckenya fibre and the ash content of the fibre reported based on dry matter analysis (Bledzki and Gassan, 1999; Lima *et al.,* 2013).

Table 1: Chemical composition of Honckenya fibre as analyzed

Description	Dry Matter (wt%)	Ash (wt%)	Extractive (wt%)	Cellulose (wt%)	H/Cellulose (wt%)	Lignin (wt%)
Sample 1	94.99	1.02	9.63	63.58	10.59	7.82
Sample 2	94.32	0.97	9.61	63..38	9.80	7.99

The analysis of Honckenya fibre showed that the composition was between those of jute, hemp, ramie and sisal that have been widely used as natural fibres in reinforcement of polymer matrices for composite materials as shown in Table 2 (Morianaa *et al.,* 2014; Poletto *et al.,* 2014; Jankauskiene *et al.,* 2015; Zhou *et al.,* 2017; Uddin *et al.,* 2019).

Table 2: Chemical composition of some selected natural fibres referenced in this study

Fibre	Cellulose (Wt%)	Hemicellulose (wt%)	Lignin (wt%)	Ash (wt%)	Extractive (wt%)	References
Jute	58-61.8	21-26	13-21	-	-	Uddin *et al.* (2019)
Ramie	73.64	13.24	1.42	1.17	-	Zhou *et al.* (2017)
Hemp	61.3-64	5.65-6.38	10.8-12.4	1.94-2.23	-	Jankauskiene *et al.* (2015)
Sisal	52.8	19.3	13.5	2.1	5.8	Lima *et al.* (2013)
Flax	77	8.7	2.8	7	-	Moriana *et al.* (2014)
Kenaf	56.6	22	11.8	1.2	-	Moriana *et al.* (2014)
Honckenya	63.48	10.20	7.91	1.00	9.62	Present study

3.2. TGA/ DTG of Untreated Honckenya Fibre

The TGA/DTG analysis of untreated Honckenya fibre was found to be thermally stable up to about 280 ºC. At this temperature, there was no significant peak in the DTG curve. There was about 3% loss in weight before a steep loss in thermal stability of up to about 80% of the total weight at a temperature of approximately 600 ºC. From the TGA thermogram, it could be observed that, there was a slight loss in weight of 0.81% at a temperature of 64.88 ºC; this could be attributed to moisture loss (Maache *et al.,* 2017). The cellulose, hemicellulose and the lignin constituents of natural fibres are known to decompose at different temperatures. Hemicellulose decomposes around 300 ˚C, while cellulose decomposes around 350 ˚C and lignin decomposes slowly from 200 ˚C to 600 ˚C. The lignin is known to be more thermally stable than hemicellulose and cellulose, therefore the higher the lignin in a natural fibre the higher the expected thermal stability (Morianaa *et al.,* 2014; Poletto *et al.,* 2014). Lima *et al.* (2013) reported that the hydrolysis of sisal natural fibre occurred in three stages- the first stage depicts endothermic dehydration of sisal fibre, the other two stages corresponding to the exothermic thermal degradation of the major constituents of the fibre. From the TGA/DTG curve of Figure 1 three major transition stages could be observed, the first stage was the endothermic dehydration of the fibre from 30 ˚C to 131 ˚C. The second stage was the exothermic decomposition of hemicellulose and cellulose with the sharp peak at 331 ˚C while the third stage was the exothermic peak which occurred at 525.73 ˚C. The observed trend was also in agreement with other reported works on TGA/DTG studies of natural fibre and their decomposition mechanism (Morianaa *et al.,* 2014;

Poletto et al., 2014; Razali *et al.,* 2015; Maache *et al.,* 2017). The TGA/DTG analysis of the untreated Honckenya fibre revealed that the fibre is suitable for reinforcing polyolefin-based composites with melting temperature up to 200 ºC, as well as thermoset plastics which are thermally cured; and most other plastics with melting point below 200 ºC.

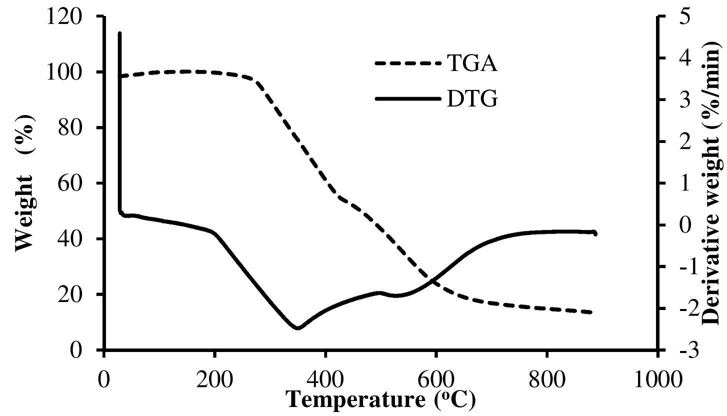

Figure 1: TGA/DTG of Honckenya fibre

3.3 FTIR Analysis of Honckenya Fibre

The spectrum for the FTIR analysis of Honckenya fibre is presented in Figure 2. The band occurring between 3753.4- 3649.74 cm^{-1} in the Honckenya indicates phenolic content present in lignin; the band at 3435 cm^{-1} corresponds to hydrogen bonded OH-stretching as reported by Morianaa *et al.* (2014). Furthermore, the band corresponding to 3322 cm^{-1} indicates the presences of cellulose in the fibre; this peak characterizes all plant based natural fibre which is consistent with the values reported by Poletto *et al.* (2014). The bands occurring around 2924 cm^{-1} with double fang is indicative of methyl and methylene found in the extractive present in the Honckenya fibre composition (Poletto *et al.,* 2014).

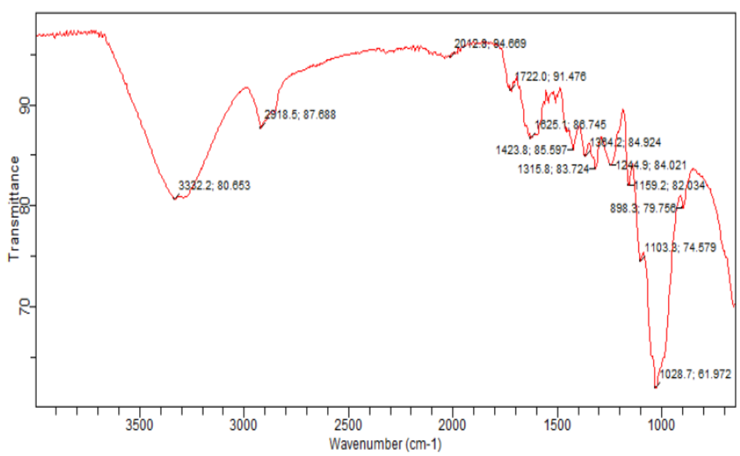

Figure 2: FTIR spectra of Honckenya fibre

The double peak present between 1726 and 1705 cm^{-1} in the untreated fibre corresponding to the carbonyl group of the hemicellulose which further confirms the presence of hemicellulose in the fibre. The band at 1509 cm^{-1} corresponding to aromatic stretching of C=C in the lignin, this peak occurs at a lower wavenumber from those of the phenolic present in the lignin. The bands that occurred between 1364.33 and 1316 cm^{-1} were attributed to the bending vibration of C-H and C-O groups in the aromatic ring in the fibre polysaccharides. The band occurring around 1300 down to around 500 cm^{-1} is the finger print region for this particular fibre (Me´sza´ros *et al.,* 2007; Fiore *et al.,* 2014; Benítez-Guerrero *et al.,* 2016).

3.4. Tensile Strength of Honckenya Fibre

The tensile strength of the untreated Honckenya fibre was 677.31±36 MPa, while the tensile modulus was 25.80±10.68 GPa and the failure strain was 2.52±0.48%. These values were compared to those of other workers who have also determined the tensile properties of some lignocellulose natural fibres that have been used as reinforcement in composites development. Table 3 gives the tensile properties of some established fibres. From the result of the failure strength, fibre tensile modulus and % failure strain, it could be seen that the Honckenya fibre has values comparable to those of the highlighted natural fibres. For instance, the tensile strength value of 677.31±36 MPa is in the range of established values for jute fibres presented in Table 3 (Bledzki and Gassan, 1999; Maache *et al.,* 2017). From the values obtained for the tensile properties, one can easily infer that the Honckenya fibre will equally be suitable for composite development, because the tensile properties is in range with those of jute, sisal, ramie, flax and kenaf that have been used in reinforcing polymer matrices (Belaadi *et al.,* 2014; Pickering *et al.,* 2016; Vaisanen *et al.,* 2017).

3.5. Density of Honckenya Fibre

The measured density of Honckenya fibre was 1.22 g/cm^3 which was found to be in range with most natural fibres that have been used in reinforcing polymer matrices. Practically, the low density of this fibre is expected to positively influence specific properties of composites made from it (Pickering *et al.,* 2016; Vaisanen *et al.,* 2017).

Table 3: Mechanical and physical properties of some selected natural fibres

Fibre	Tensile Strength MPa)	Tensile Modulus (GPa)	Failure Strain (%)	Density (g/cm^3)	References
Jute	393-800	10-55	1.5-1.8	-	Pickering *et al.* (2016)
Ramie	400-938	44-128	2-3.8	1.17	Pickering *et al.* (2016)
Hemp	550-1110	58-70	1.6	1.94-2.23	Pickering *et al.* (2016)
Sisal	507-855	9.4-28	2-2.5	2.1	Pickering *et al.* (2016)
Flax	345-1830	27-80	1.2-3.2	7	Pickering *et al.* (2016)
Kenaf	930	53	-	1.2	Vaisanen et al., 2017
Honckenya	677.31±36	25.80±10.68	2.52±0.48	1.22	Present Study

3.6. Weibull Parameter Estimation

Figure 3 shows the line fit plot for the fibre, the coefficient of correlation R^2 was 0.98 which indicates a good fit between the experimental values and the predicted values based on linear least square regression analysis. From the slope of the line fit plot the Weibull modulus (m) could be estimated, while substituting into the transformed two parameter Weibull equation the characteristic strength (σ_o) can be calculated (Belaadi *et al.,* 2014; Fiore *et al.,* 2014; Maache *et al.,* 2017). The Weibull modulus was 2.26 which showed that the fibre failure is a brittle type as well as revealing information on the scatter of the result. The m value obtained for the untreated fibre indicates that the measured failure strength of the fibre has a large deviation (Thamae and Baillie, 2007). Also, the characteristic strength was 683.66 MPa for the untreated Honckenya fibre. This value as obtained from the Weibull distribution function is an indication of the inherent failure stress of the fibre (Belaadi *et al.,* 2014). One of the main features of weibull distribution is based on the fact that it could predict material failure and reliability; this study exploited Weibull capability to estimate survival probability of the untreated Honckenya fibre.

Figure 3: Line fit plot of Honckenya fibre

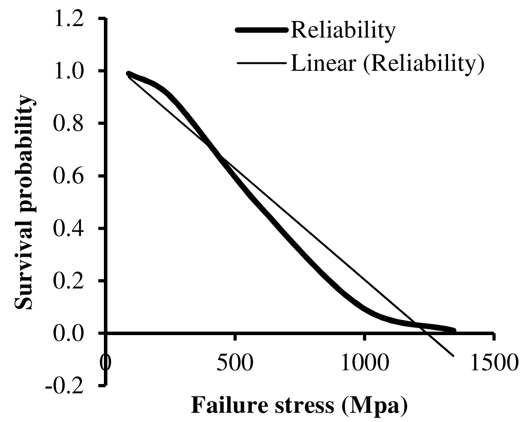

Figure 4: Survival Reliability plot of Honckenya fibre

The reliability plot for the fibre is shown in Figure 4. The failure stress of the fibre is plotted against fibre survival probability, and this gives an indication of the fibre reliability. The reliability plot evaluated from the Weibull distribution indicates that for any given mass of the Honckenya fibre bundles, the stress distribution within the fibres suggests that only 50% of the fibre would have a failure stress of 581 MPa and above, while only 10% of the fibre mass could survive a failure stress of 988.70 MPa; moreover, 80% of the fibre would survive a failure stress of 352.12 MPa and below which is an indication that the bulk of the fibres fall within the expected fibre failure strength range as highlighted in Table 3.

4. CONCLUSION

From the study, Honckenya fibre was characterized to determine its physical, chemical, mechanical and thermal properties. The suitability of Honckenya vegital bast fibre as reinforcement in polymers was established. It could be inferred that the fibre is suitable for reinforcing polymer matrix. With the following conclusions drawn from the study:

i. The TGA gives the onset of thermal degradation as 280 ^{o}C at 3% weight loss.

ii. The fibre composition analysis showed that Honckenya have cellulose content of 63.48±0.1414wt%, while the hemicellulose content was 10.195±0.5886wt% and the lignin content was 7.908±0.1202wt%.

iii. The finger print and the functional groups present in the fibre at different absorption bands for the fibre was established from the FTIR spectrum. Cellulose occurred at 3322 cm^{-1}, hemicellulose bands occurred at 1726 cm^{-1} and 1705 cm^{-1} while lignin occurred at 1509 cm^{-1} bands.

iv. The density of the fibre was determined to be 1.22 g/cm^3.

v. The fibre failure stress was determined as 677.31±359.08 MPa, the modulus was measured as 25.80±10.68 GPa and the failure strain as 2.52±0.48%. these properties are in range of most common natural fibres that have been used for polymer applications.

vi. From the Weibull distribution the Honckenya reliability indicates that at least 50% of the fibre would have a failure stress of 581 MPa and above.

5. ACKNOWLEDGMENT

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6. CONFLICT OF INTEREST

There is no conflict of interest associated with this work.

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