BOOK OF PROCEEDINGS
(Available at: http://acsnigeria.org/publications/proceedings)

Characterization and Reinforcement of *Zea mays* Fibre in Geopolymer Composite

Grace O. Ayorinde, Oluwadurotimi O. Akintade, Olapeju D. Folaranmi, Fausat O. Odujebe
Department of Chemical Sciences, Faculty of Applied Sciences, KolaDaisi University, Ibadan, Oyo State, Nigeria.

Corresponding Author's email: tsnakin@gmail.com

ABSTRACT

For technical, economic and environmental reasons, the construction industry now widely accepts the development of composites using natural fibres and eco-friendly materials in structural concrete. The goal of this study was to create a composite made from cornstalk (*Zea mays*) fibre, an agricultural waste and geopolymer. The *Zea mays* fibre was extracted and characterized for its physical properties and chemical composition. The geopolymer precursor, meta-kaolin, was activated using an equal volume of sodium silicate and 12% sodium hydroxide. The impact of the cornstalk fibre sizes (0.425mm and 1mm) and fibre volume (1%, 2.5%, 5%, 7.5%, and 10%) on the compressive strength of the reinforced geopolymer composite were investigated. The mean values obtained for the physical and chemical properties of cornstalk fibre were 10%, 20.5%, 25.5%, 45.3 %, 11.5 %, 0.39 gcm^{-3}, 35.3% and 49.67% for moisture content, cold water solubility, hot water solubility,1 % NaOH, alcohol-benzene solubility, density, lignin content and cellulose content. The mean values obtained for compression strength of geopolymer composite reinforced with 0.425 mm corn stalk fibre ranges from 15.527 N/mm^2 – 38.80 N/mm^2 and for 1.00 mm it ranges from 18.02 N/mm^2 – 41.74 N/mm^2. The results revealed that an increase in cornstalk fibre improves the compressive strength of the geopolymer composite concrete and that the higher mechanical strength was observed with geopolymer reinforced with 1.00 mm fibre size. In conclusion, the findings show that the cornstalk fibre were suitable for reinforcement.

KEYWORDS: *Zea mays*, Composite, Geopolymer, Reinforcement, Compressive strength.

1. INTRODUCTION

The development of composites using natural fibre and more eco-friendly matrices has been prompted by the demand for energy-efficient building and industrial components. Composites exist in nature. Composites are materials that consist of two or more materials that are combined in a way that allows the materials to stay distinct and identifiable. The goal of composites is to allow the new materials to have strengths from both materials, often covering the original materials' weaknesses[1]. The two constituents of composites are matrix and reinforcement materials. The matrix adopted in this study is geopolymer made from meta-kaolinite and its activator, sodium silicate and sodium hydroxide and the reinforcement material is Corn (*Zea mays*) stalk waste fibres.

Corn (*Zea mays*) waste fibres are lignocellulosic fibres consisting of corn cob, corn husk, corn stalk and corn stover, often discarded[2, 3]. Physio-chemical properties of the lignocellulosic fibres from different sources are compared. Studies on lignocellulosic fibre-reinforced bio-plastic composites and state-of-the-art fibre-reinforced composites are reviewed[4]. The preparations of the fibres to form nano-fibril, cellulose nano-fibril and lignin-containing cellulose nano-fibril are also discussed.

Along with this, issues to improve fibre-plastic matrix compatibility through mechanical disintegration and surface modification treatment on fibre are also reported [5, 6]. Further treatment of the fibres could improve the composite properties for various applications[7]. In this study, the aim is to create a composite made from cornstalk (*Zea mays*) fibre, an agricultural waste reinforced with a geopolymer; also, to determine the functional group in *Zea mays* fibre using FTIR.

2. MATERIALS AND METHODS

2.1. Sample collection and preparation

Dry cornstalks used were collected on a farm at Saki town, Agosuru off Irawo road Agbele in Oyo State, Nigeria. The husks, leaves and inner part (pitch) were manually removed from the stalk's outer part, which was grinded into smaller particle sizes. The geopolymer precursor (kaolin) used was collected from Mile 7, Abeokuta, Ogun state, Nigeria. The kaolin was calcined at 600^{o}C for three hours from dehydroxylation to meta-kaolinite[7]. The pure geopolymer process was done by activating the precursor with an equal volume of sodium silicate and sodium hydroxide while sodium hydroxide concentration was varied (4%, 8%, 12% and 16%). A geopolymer of 12% concentration was observed with the highest compression strength and this percentage of sodium hydroxide was adopted for the subsequent composite fabrication[8].

2.2. Determination of Physical and Chemical Properties

Moisture content, cold water solubility, hot water solubility, 1% NaOH, alcohol-benzene solubility and density were determined using ASTM standard. The lignin and cellulose content were also obtained using TAPPI and Kushner-Hoffer methods respectively[9].

2.3. Geopolymerization process and composite formation

Kaolin was calcined at 700^{o}C for 3 hours to convert it to meta-kaolinite. 160 g of the meta-kaolinite was activated using a mixture of NaOH and commercial grade of Na_2SiO_3 in a ratio 1:1. For pure geopolymer, the percentage of NaOH was varied at 4%, 8%, 12% and 16% respectively. Pure geopolymer activated with 12% NaOH was observed with the highest value of compression strength and this percentage of NaOH was adopted for subsequent composite fabrication. 1.6 g of the 425 mm mesh size of cornstalk fibre was thoroughly mixed with 158.4g of meta-kaolin (1% fibre of 160g composites). The mixture was activated using mixture of 12% NaOH and commercial-grade Na_2SiO_3. The mixture was transferred into a wooden mould of 50 mm x 50 mm x 50 mm and left for 48 hours. The moulded bricks were made to pass through ambient curing for 10 days with 24 hours interval check of loss in weight. Compression strength of the brick was determined. The experiment was repeated for 2.5%, 5.0%, 7.5% and 10% of cornstalk fibre for the same size and also for 1.00 mm fibre size respectively[9,12]. The general experimental procedure is illustrated in Figure 1.

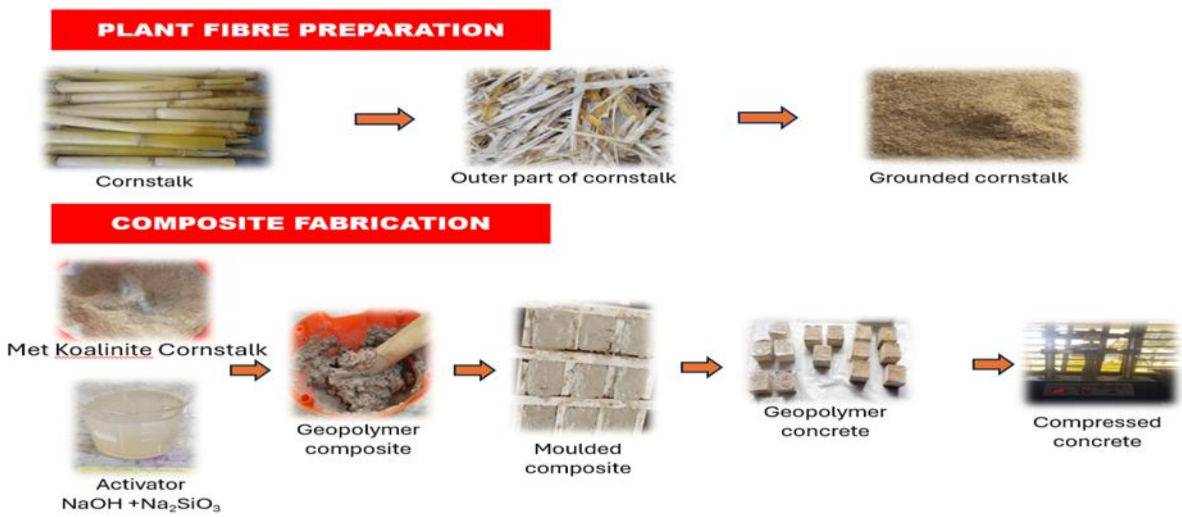

Figure 1: Sample Preparation and Treatment of Cornstalk Fibre

3. RESULTS AND DISCUSSION

Physical properties such as density, moisture content, hot and cold solubility, lignin content, cellulose content, 1% NaOH solubility were analysed to compare the corn stalk fibre with other lignocellulose fibre. Finally, the effect of the reinforcement was observed by carrying out the compressive strength of the composite concrete fabricated.

Table 1: Mean values obtained for Physical Properties of *Zea mays* stalk fibre

Lignocellulose fibre	Moisture contents (%)	Cold-water solubility (%)	Hot-water solubility (%)	1 % NaOH solubility (%)	Alcohol-benzene solubility (%)	Density (g/cm^3)
Zea mays stalk	10.00±0.50	20.50±2.18	25.50±2.50	45.30±2.52	11.50±1.73	0.39±0.03

Table 1 above, present the mean values obtained for physical properties of *Zea mays* fibre. The result obtained were in accordance with the result reported by Duraisamy *et al*., 2020[10]. According to literature, good lignocellulose fibre is expected to have low moisture content and low extraneous particles. The incorporation of fibre with considerably low extraneous particle in cementitious material composites as a reinforcement can enhance the flexural limit during splitting, durability, ductility, and break resistance compared to the unreinforced matrix[11].

Table 2: Mean values obtained for Chemical Properties of *Zea mays* stalk fibre

Lignocellulose fibre	Cellulose contents (%)	Lignin contents (%)
Zea mays stalk	49.67±0.58	35.30±3.51

Table 2 above, presents the mean values obtained for the chemical properties of *Zea mays* fibre. The results obtained were in accordance with the results reported by Okanlawon et al., 2022[9]. According to literature, good lignocellulose fibres are expected to have high cellulose content and low lignin content. The higher the cellulose content the better the plant for composite reinforcement[9].

Table 3: Mean values obtained for Compression test of Pure Geopolymer

Percentage of NaOH (%)	Volume ratio of NaOH to Na_2SiO_3	Ratio of Meta-kaolinite to Activator	Compression strength (Nmm^{-2})
4	1:1	2:1	9.07±3.80
8	1:1	2:1	8.93±3.40
12	1:1	2:1	10.93±1.89
16	1:1	2:1	3.20±2.78

Table 3 present the mean values obtained for compression strength of pure geopolymer. The result for compression strength of the pure geopolymer ranges from 3.20 Nmm^{-2} to 10.93 Nmm^{-2}. The pure geopolymer activated using 12 % NaOH was observed with the highest compression strength and this percentage of NaOH was adopted for subsequent geopolymer composites.

3.1. FTIR Characterization of cornstalk fibre

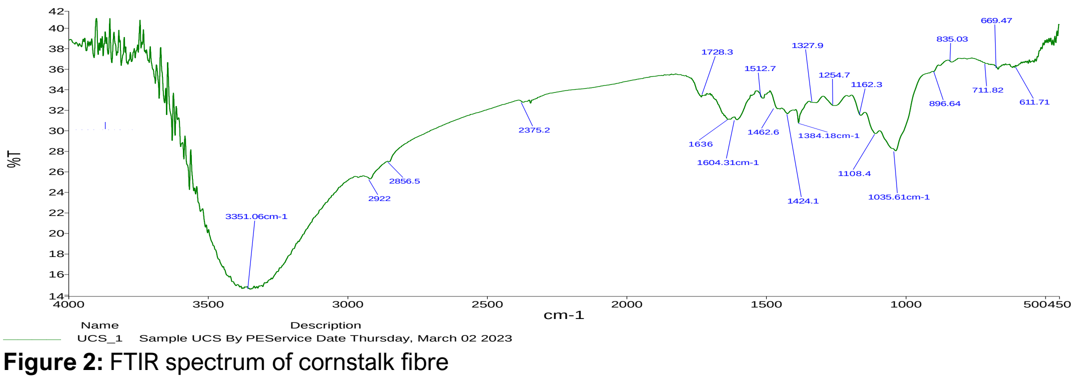

Figure 2: FTIR spectrum of cornstalk fibre

The FTIR spectrum of cornstalk fibre is shown in Figure 2. According to the spectrum, there is broad and strong band at 3351.06 cm^{-1} which could be attributed to –OH stretch of alcohol. Also, from the spectrum, there are sharp and narrow bands at 2922 cm^{-1} and 1728.3 cm^{-1}, assigned to $-CH_2$ pyranose structure and C=O carbonyl of cellulose respectively. Another prominent band in the functional group region were observed at 1636 cm^{-1} and 1604.31 cm^{-1}, which could also be attributed to H-O-H group. These bands observed in this spectrum confirmed the cellulose structure in cornstalk fibre which is common to all lignocellulose fibres and is in accordance with the result reported by[10].

Table 4: Mean values obtained for Compressive strength of geopolymer composite

Fibre size (mm)	Fibre volume (%)	Ratio of Fibre to geopolymer	Compression strength (Nmm^{-2})
0.425	1 %	1:150	15.52±0.92
0.425	2.5 %	1:60	19.46±0.95
0.425	5.0 %	1:30	22.12±1.13
0.425	7.5 %	1:20	26.65±1.05
0.425	10 %	1:15	38.80±1.19
1.00	1 %	1:150	18.02±0.72
1.00	2.5 %	1:60	20.91±1.03
1.00	5.0 %	1:30	25.53±0.74
1.00	7.5 %	1:20	29.42±2.03
1.00	10 %	1:15	41.74±0.48

Table 4 shows the results of the mechanical properties obtained from geopolymer composite reinforced with cornstalk fibre of different size and volume. The result indicates that the compression strength of the composite ranges from 15.52 Nmm^{-2} to 41.74 Nmm^{-2}. These results were in agreement with the result reported by[11]. According to the results the higher the ratio of fibre to geopolymer, the higher the compression strength of the geopolymer. However, the higher mechanical strength is observed with geopolymer reinforced with 1.00 mm fibre size compare to geopolymer reinforced with fibre size of 0.425 mm[4]. Figures 3 and 4 shows the curing time of reinforced geopolymer with different NaOH percentage, reinforced geopolymer with 0.425 mesh size and 1nm mesh size cornstalk fibre respectively. The figures shows that it took 7days for reinforced geopolymer with cornstalk fibre to cure, which indicates that as the days increases the weight of the bricks reduce drastically until it reach the point where the reduction difference is very low.

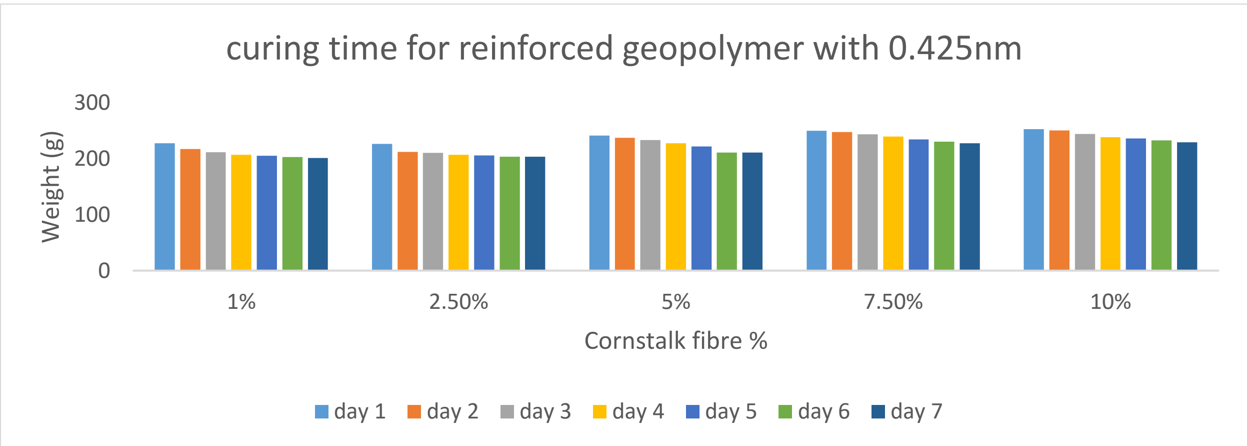

Figure 3: Curing time for reinforced geopolymer with 0.425nm mesh size cornstalk fibre

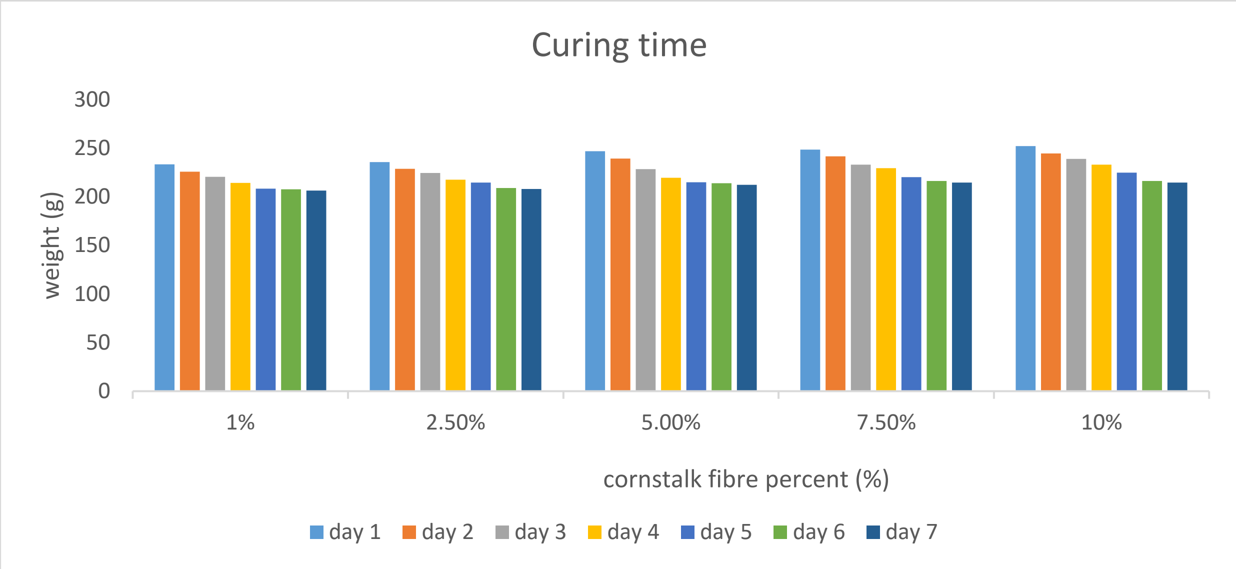

Figure 4: Curing time for reinforced geopolymer with 1nm mesh size of cornstalk fibre:

4. CONCLUSION

Corn stalk from agricultural waste can be utilized to create cornstalk fibre. The compressive strength of polymer composites with lignocellulose fibre are significantly affected by the properties of their components, the quality of bonding between the geopolymer and the lignocellulosic material. The characterization and compressive strength of corn stalk fibres revealed that they may be used as reinforcement for friction composites, which is an environmentally beneficial method of utilizing waste maize stalks. The usage of natural fibre is ought to be made more common. A code of practice should be created after looking at all the related issues to the reinforcement of natural fibre.

ACKNOWLEDGMENTS

The authors appreciate the Chemical Sciences Laboratory of KolaDaisi University, Ibadan

REFERENCES

(1) Mohammed, L., Ansari, M. N., Pua, G., Jawaid, M., & Islam, M. S. A review on natural fibre reinforced polymer composite and its applications. *International journal of polymer science*. **2015,** 1-15. http://dx.doi.org/10.1155/2015/243947.

(2) Liu, Y., Wang, L., Liu, D., Ma, Y., Tian, Y., Tong, J., & Saravanakumar, S. Evaluation of wear resistance of corn stalk fibre reinforced brake friction materials prepared by wet granulation. *Wear*, **2019,** *432*, 102918.

(3) Eloise K. Billington, Theona S̡ & Michael P. Shaver. Mechanical Properties and Recyclability of Fibre Reinforced Polyester Composites. ACS Sustainable Chem. Eng. **2024,** 12, 10011−10019.

(4) Bongarde, U. S. and Shinde, V. D. Review on natural fibre reinforcement polymer composites. *Int. J. of Eng. Sci. and Inn. Tech.*, **2019,** *3*(2), 431-436.

(5) Rodriguez, M., Rodriguez, A., Bayer Resplandis, J., VilasecaMorera, F., GironèsiMolera, J., & MutjéPujol, P. Determination of corn stalk fibres' strength through modelling of the mechanical properties of its composites. *BioRes., **2010,*** 5(4): *2535-2546*.

(6) Anurag Sharma and Sunil C. J**.** Fibre-Reinforced Polyurethane Matrix Composites for Engineering Applications. *Polyurethanes: Preparation, Properties, and Applications Volume 1: Fundamentals*. **2023.** 6:101-118. doi**:** 10.1021/bk-2023-1452.ch006

(7) Mansourghanaei, M., Biklaryan, M., & Mardookhpour, A. Experimental Investigation of the

Effect of Nanosilica on the Mechanical Properties and Durability of Slag Geopolymer Concrete. *J. of Con. Struc. and Mat.*, **2022,** *7*(1), 76-89.
(8)Alshaaer, M. (2013). Two-phase geopolymerization of kaolinite-based geopolymers. *Applied clay science*, *86*, 162-168.
(9) Okanlawon, F. B., Olaoye, K. O., Awotoye, O. O., Adegoke, A. O. Fibre characteristics and chemical composition of *Delonix regia* (Boj. Ex Hook.) Raf. Wood. *J. of Res. in For., Wild. & Env.*, **2022,** 14(3).
(10) Duraisamy, R., Shuge, T., Worku, B., Berekete, A. K. and Ramasamy, K. M. Extraction, Screening and Spectral Characterization of Tannins from Acacia Xanthophloea (Fever Tree) Bark *Res. J. of Tex. and Lea. (RJTL).* **2020,** 1(1), 1-12
(11) Ahmad, R., Hamid, R., and Osman, S. A. Physical and Chemical Modifications of Plant Fibres for Reinforcement in Cementitious Composites. *Hindawi Adv. in Civil Eng.* **2019,** 5185806, 1-18. https://doi.org/10.1155/**2019**/5185806.
(12) Biljan, R. I., Aleksandra, A. M., and Ljiljana, T. M. Thermal Treatment of Kaolin Clay to obtain MetaKaolin. Inst. For Testing Mat. **2010,** 64(4), 351-356. https://doi:10.2298/HEMIND1003220141