


ORIGINAL RESEARCH

Sustainability

Synthesis, characterization, physicochemical, and electrical properties of natural (bio) nanofluids

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Abstract

Energy conservation and sustainability to reduce the dependence on conventional sources have resulted in modified or advanced process practices. One such is the use of nanofluids for enhanced energy efficiency. However, such practices must not be at the cost of environmental hazards. The current study emphasizes bio-based nanofluids formulated at five different volumetric concentrations (0.2%, 0.4%, 0.6%, 0.8%, and 1.0%) using Flamboyant (Royal Poinciana) tree bark nanoparticles with ethylene glycol as base fluid. The nanoparticles synthesized by cost-effective extensive ball milling technique were spherical in shape. Analyzing the nanofluid with TEM confirms the particles as evenly distributed with an average diameter of 26 nm. Elemental analysis shows that the bio powder contains oxides of Calcium and Silicon. The pH, electrical conductivity, and viscosity of the prepared flamboyant tree bark-ethylene glycol (FTB-EG) nanofluid were quantified between 20 and 70°C. Although the properties enhanced with increase in concentration, the viscosity and pH decreased with temperature rise, while the electrical conductivity behaved contradictory. The maximum and minimum values of the properties were attributed to 1.0% and 0.2% concentrations, respectively. The correlations were proposed and the deviation between the measured and correlation data was less than 10%.

KEYWORDS

bio-nanofluids, *Delonix regia*, electrical conductivity, pH, viscosity

1 | INTRODUCTION

Since nanofluids exhibit enhanced thermal properties employing in thermal equipment used in various industrial processes can operate more effectively, producing sustainable energy, unit miniaturization, and cost reductions.^{1–6} However, the dangers that poses by

synthesizing and employing nanofluids to health, socio-economic and the environment lack complete analysis and understanding.⁷ In this context, the demand for creating ecologically friendly green nanofluids has been more crucial in tandem with the booming exploration of nanofluids. Green initiatives involve reducing environmental risk, conserving natural resources, and implementing sustainable practices

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that lessen the detrimental effects of human activity.⁸ In this respect, green nanotechnology uses environmental resources to reduce or eliminate environmental concerns associated with using any conventional metallic or non-metallic nanoparticles and encourage the progressive substitution of traditional and unharmed nanomaterials. Until now, the research on nanofluids produced using environmentally benign techniques had a limited impact.⁹

Green nanofluids' thermo-physical characteristics have recently been enhanced by adding biomaterials, including parts of plants or trees in powdered form, to the base fluid. However, only a handful of research efforts have been made on exploring green nanofluids. For instance, research like the one by Meka Chufa et al.¹⁰ used *Vernonia amygdalina* plant leaf extract to reduce graphene oxide and sustainably produce graphene. Terpenoids and polyphenols served as the plant extract's reducing and capping agents. They believed that making stable nanoparticles using the carbonyl and hydroxyl groups was the most effective strategy for producing graphene-based particles on a wide scale. Investigators are facing constraints, particularly in the biomedical field, because the reducing agents and stabilizers that are typically used to generate nanoscale materials and enhance their stability by altering their surface tension harm the surroundings.¹¹ In this context, it is critical to switch to safer reagents derived from plants, such as photochemicals, gum, isoflavones, organic acids, and catechins to substitute hazardous substances and stabilizers.¹²

Rangaraj and Venkatachalam¹³ produced Green silica powders from the bamboo's residual leaf biomass, and its toxic behavior was assessed by an in vitro experiment. They also emphasized that employing those leaves to make silica nanoparticles on a big scale is preferable to adopting chemical precursors of silica for medication administration and applications.

Khdher et al.¹⁴ revealed the green based alumina nanofluids to be a harmless and compostable bio-glycol produced from plants. Despite their nanofluids' yielded extended stability without requiring any surfactant. Using the identical proportion of bio-glycol, EG, and PG, they reported an increased thermal conductivity. The thermal conductivity enhancement with bio-glycol was twice that of EG and quadruples times that of PG. In a different study Abdolbaqi and co-authors¹⁵ were able to disperse Al₂O₃ nanoparticles in water and bio-glycol at two different ratios (60:40; 40:60). They outweighed the thermal conductivity of PG and water blend at the same proportions by 7.5%. The thermal conductivity of SiO₂ and TiO₂ nanofluids with bio-glycol and water-blended base fluid was also determined.^{16,17} At the same time, they compared the PG and water blend at the same proportions and found an increase in TiO₂ properties by 12.6% and in SiO₂ properties by 7.2%. In the last few years, studies have emerged on investing in the properties of green nanofluids prepared from the powder form of plant species like banana fiber, mango bark, and palm kernel fiber.^{18–20}

Nevertheless, research on green nanofluids is in the early stages of progress. It is challenging to create them from non-toxic and sustainable resources since additional research is required to determine the suitability, sustainability, and accessibility of the resources. The actual understanding of green nanofluids in the

literature²¹ also brings on discrepancies and controversies. A significant share of the literature emphasizes the need to make nanoparticles through safe green processes rather than risky traditional ones. The creation of sustainable projects for the product of nanofluids encourages conservation by utilizing natural resources. However, their deployment in the long term is fraught with challenges that are yet highly debated, underscoring the urgent need for more research on the topic.

Based on all the above said, the following investigations are considered as the first stage of research on PDRA, considered novel green materials contributing for potential fluids across heat exchangers, radiators, and coolants across cutting tool processes.

- I. Synthesizing bio nanoparticles from Flamboyant (*Delonix regia*) Tree Bark using a cost-effective and environmentally friendly method.
- II. Synthesizing and stabilizing the nanofluids at different volumetric concentrations using EG as base fluid.
- III. Characterizing the nanopowder and nanofluid using the size and constituent-finding equipment.
- IV. Quantifying the properties such as pH, viscosity, and electrical conductivity of the nanofluids at different temperatures.
- V. Developing correlations for the viscosity and thermal conductivity of these green nanofluids based on the retrieved data.

2 | MATERIALS AND METHODS

2.1 | Synthesis of nanopowder

About 100 kg of flamboyant tree bark (FTB) was collected and sun-dried for about 4 weeks and then desiccated with an oven at a temperature of 40–50°C to confirm the removal of the entire residue moisture. The dried Flamboyant tree bark was crushed mechanically to produce macrofibre fragments. It was again loaded into the ball mill for further breakdown to smaller sizes. The charged ball mill was left to run for 48 h uninterrupted. These were again ball milled for 24 h using a THYREC ball-milling machine with 63 steel balls of 14 kg each and 66 rpm speed.

2.2 | Nanofluid preparation

Based on the chosen volume fractions (0.2%, 0.4%, 0.6%, 0.8%, and 1.0%), respective masses were measured using a sensitive electronic weighing machine (RADWAG AS 220-R2; accuracy ±0.01 g). The nanomaterials were dispersed into 100 mL of the base fluid which is ethylene glycol (EG). To homogenize the mixture, magnetic stirring (Hotplate Stirrers, Ha400 HSB, Indonesia) at 600 rpm for 10 min was introduced. The homogenous mixture maintained at 20°C using a thermal bath (LAUDA PROLINE RP1840) was then undertaken to intensive ultrasonic agitation, high-shear mixing, and homogenizing using a probe sonicator (QSonica ultrasonicator-Model Q-700) set at

amplitude 50% with pulse on and off set for 5 and 2 s, respectively. Five samples of 0.2% concentrated nanofluids were sonicated at different periods (10–50 min) to identify the optimal sonication time by evaluating the stability by sedimentation. The minimum sedimentation of the nanoparticles was noted for the sample ultrasonicated for 30 min.

2.3 | Instrumentations for sample and property characterization

The morphology of the dispersed particles was analyzed by Scanning Electron Microscopy (SEM) (Zeiss Crossbeam 540). However, the even distribution of the particle as well as the particle interaction with the base fluid is observable using a Transmission Electron Microscope (TEM) (JEOL JEM-2100F) imaging.

The average density of the dispersed nanoparticle was determined by careful compaction of a known volume of the powder into a pellet and is weighed using a sensitive electronic weigh balance. The sample nanofluids properties such as viscosity, pH, and electrical conductivity were measured similarly to one of our previous studies²² using an SV-10 device (sine wave viscometer with $\pm 3\%$ accuracy, A&D, Japan), Jenway 3510 pH meter (± 0.003 accuracy and 2–19.999 range), and EUTECH (CON700) electrical conductivity meter ($\pm 1\%$ accuracy) instruments, respectively. All the instruments were calibrated before being put to use for experimental study. The viscometer was calibrated by testing it with the base liquid and comparing the data with ASHRAE values. The pH meter was calibrated with three liquids of pH 4, 7, and 10 supplied by the supplier. The conductivity meter was calibrated with a standard reference liquid ($1413\text{-}\mu\text{S}/\text{cm}$) at 25°C per the manufacturer's specifications.

3 | RESULTS AND DISCUSSION

3.1 | Nanopowder and nanofluid characterization

The powder form of the tree bark is shown in Figure 1. The SEM images in Figure 2 represent the morphology of the powder, which confirmed spherical shape in an agglomerated state. However, the average particle size of the powder is about 100 nm. This cannot be considered the exact measure of dimensions as it is just a part of the sample that has been considered for morphology analysis. A precise analysis was also done through TEM after dispersing the powder into the base fluid to confirm the particle size.

The TEM image in Figure 3 represents the hydrodynamic interaction between the powder and the base fluid, evident from the darkened encapsulation zone around the powders. In addition, the TEM image demonstrates good distribution of particles within the base fluids (EG). The average particle size of 26 nm was notable from TEM images. The density of the nanopowder was $0.4648\text{ g}/\text{cm}^3$ at an uncertainty of $\pm 0.79\%$. The elemental analysis results depicted in Figure 4 showed higher traces of oxygen followed by calcium and silicon indicating the presence of oxides of calcium and silicon.

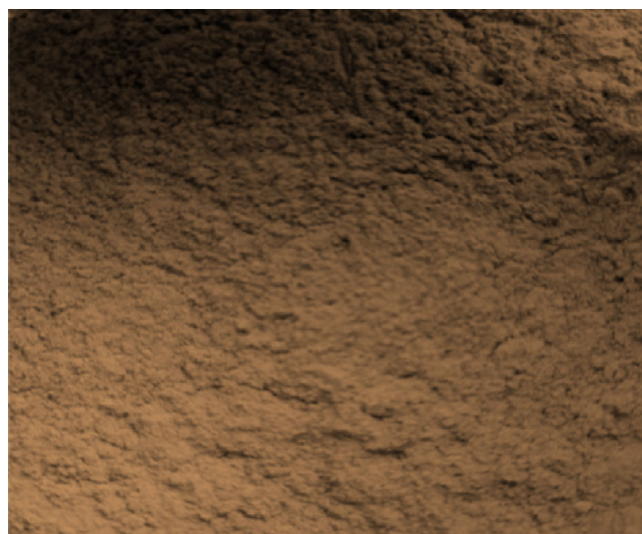


FIGURE 1 Image of FTB in powder form.

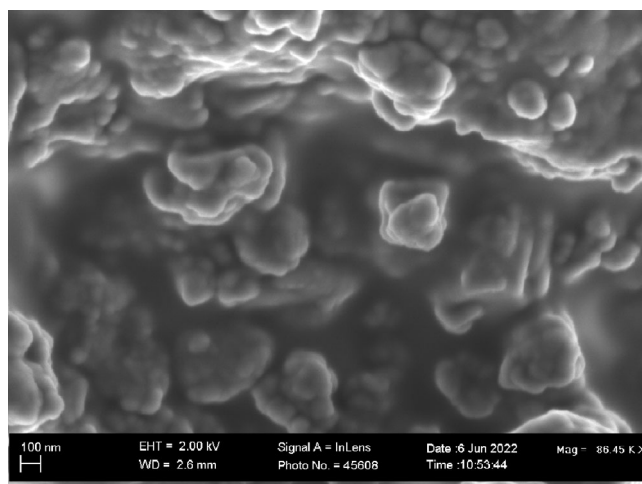


FIGURE 2 SEM image of FTB nanoparticles.

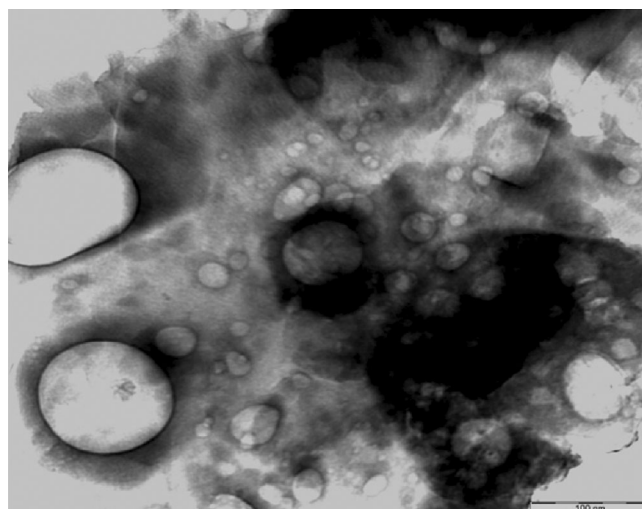


FIGURE 3 TEM images of FTB nanoparticles.

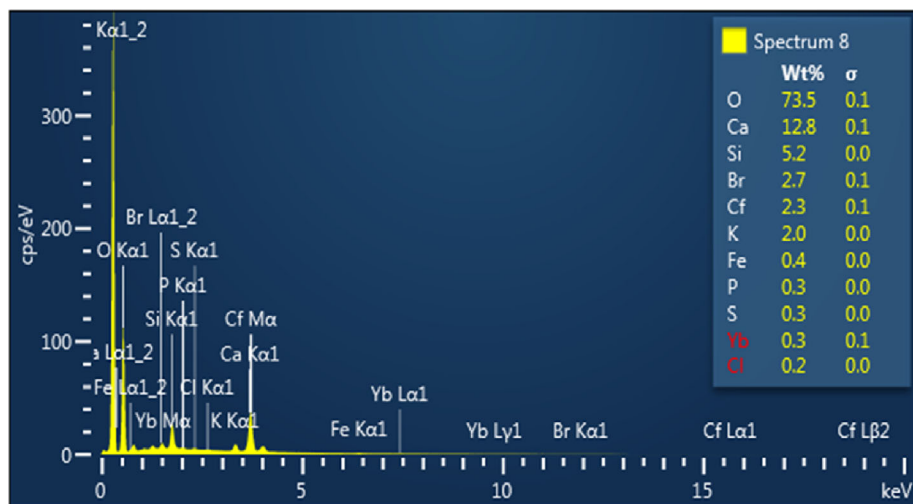


FIGURE 4 Result of elemental analysis of FTB nanoparticles.



FIGURE 5 Images of the prepared natural (bio) nanofluids.

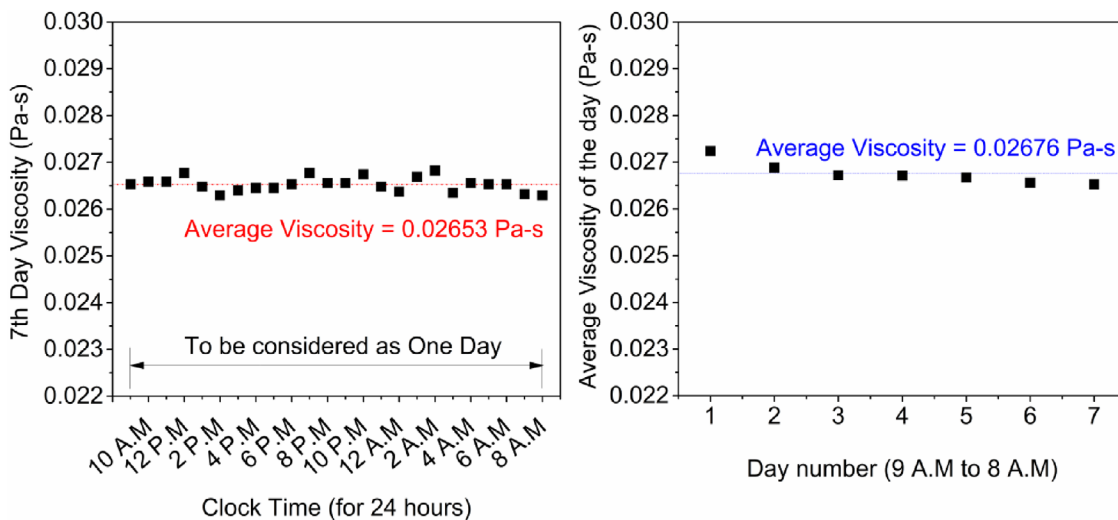


FIGURE 6 Stability evaluation by transient viscosity approach.

3.2 | Stability of the nanofluid

Besides knowing about the even distribution of particles within the base fluid from the TEM images, it is a must to quantify the nanofluid stability by two general methods: (a) visual inspection and (b) transient viscosity method.

Although that there was literally no sedimentation of particles for 48 h notable from visual inspection of the prepared bio-nanofluids

(Figure 5), the transient viscosity method (Figure 6) employed for a week supports the claim that the sample nanofluids are stable without addition of any surfactant. It might also be evident from the TEM image that the fluid layer has encapsulated around the particle surface, signifying their affinity for better-stabilized states. Besides, EG which is also viscous in nature, is also be a reason for the prolonged stability of the nanofluid. However, the fact must be the biocompatible capping agents available naturally within the bio powder that

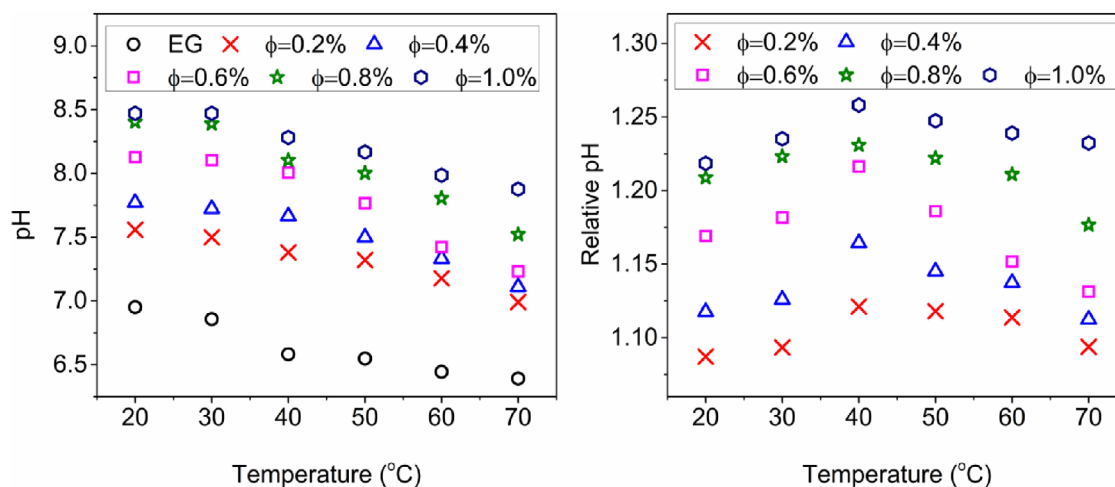


FIGURE 7 pH and its enhancement of FTB-EG nanofluid under varied concentrations and temperatures.

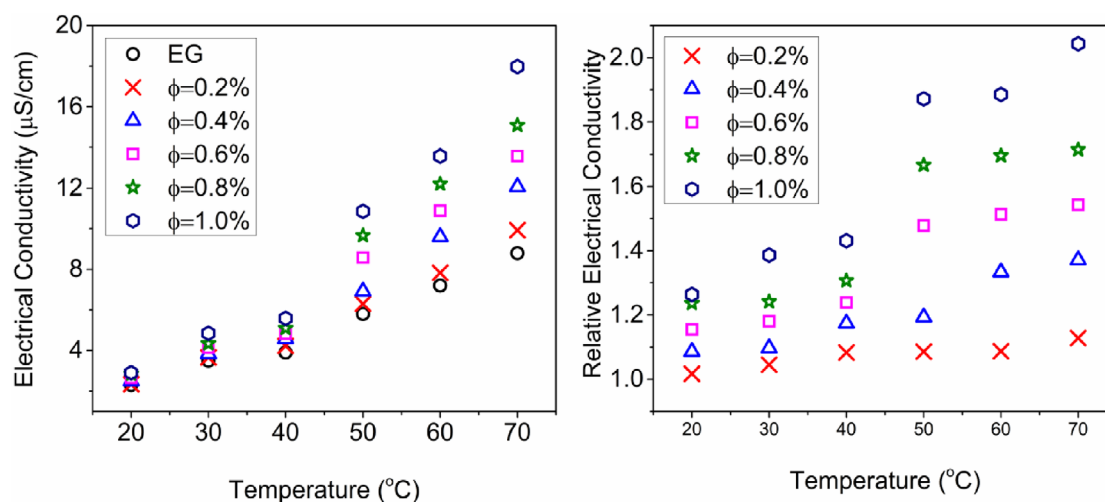


FIGURE 8 Electrical conductivity and its enhancement of FTB-EG nanofluid under varied concentrations and temperatures.

might develop an electrostatic repulsion preventing the particles from agglomerating after being suspended in the fluid (i.e., EG).

3.3 | Effect of nanoparticles concentration and temperature on the pH of nanofluids

Figure 7 shows how temperature influences the pH of the FTB-EG nanofluid for different volumetric concentrations. The FTB nanoparticles' constituents play a role in the nanofluids' pH change. For example, CaO, which is a significant part of this bio-product, is generally alkaline in nature. It might be the reason for an increase in the pH of the nanofluids at room temperature in comparison with pure EG. Further an increase in concentration of FTB nanopowders, enhances the pH of the nanofluid. However, the drop in pH when the temperature was raised from 20 to 70°C must be significant due to the disassociation of flavanoids, saponins, and tannins in the bio powders. These

phytochemical constituents generate a pH between the range of 3 and 5.5, thus reducing the overall pH of the nanofluids. Besides, the temperature modifies the pH by disassociating OH^- from the base fluid. The base fluid pH dropped as the temperature was increased from 20 to 70°C. Nevertheless, there must be a temperature condition until the pH reduction by the phytochemical constituents is annulled by the OH^- ions from dissociated glycols. Such a scenario is evident in Figure 7, where the relative pH turns beyond 40°C.

3.4 | Effect of nanoparticles concentration and temperature on the electrical conductivity of nanofluids

Electrical conductivity, which is the measure of total ions in the liquid, is connected with the pH of the fluid. It is notable from Figure 8 that the addition of this nanoparticle did not cause a drastic effect on the

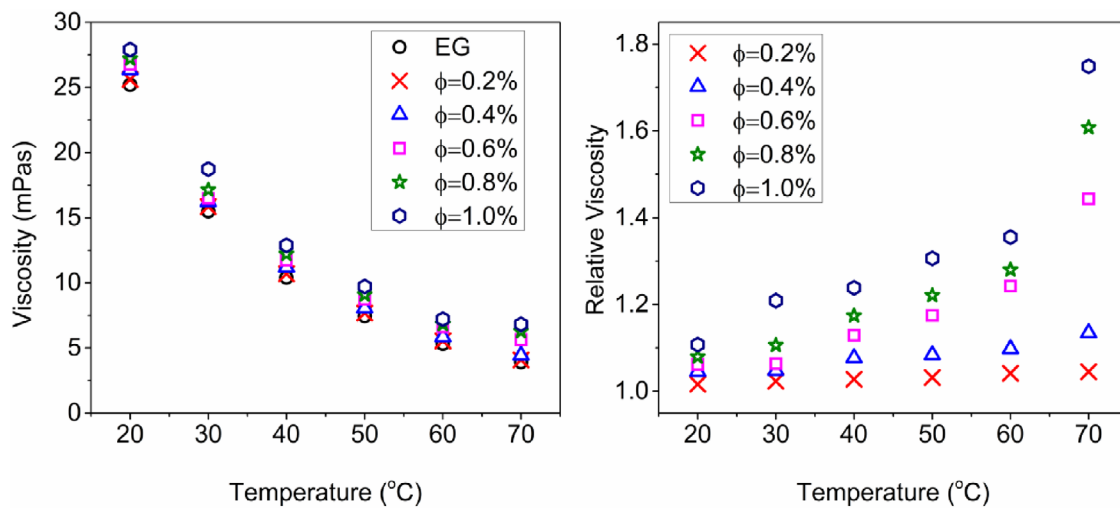


FIGURE 9 Viscosity and its enhancement of FTB-EG nanofluid under varied concentrations and temperatures.

TABLE 1 Values for correlation constants.

Parameter	A ₁	A ₂	A ₃	A ₄	A ₅	A ₆	A ₇
Relative pH	0.885	0.410	7.969 × 10 ⁻³	-5.003 × 10 ⁻³	-0.110	7.650 × 10 ⁻⁵	4.317 × 10 ⁻⁵
Relative electrical conductivity	1.018	-0.165	-1.623 × 10 ⁻³	1.677 × 10 ⁻²	8.069 × 10 ⁻²	4.476 × 10 ⁻⁶	6.995 × 10 ⁻⁶
Relative viscosity	1.138	0.186	-9.439 × 10 ⁻³	3.357 × 10 ⁻³	-0.189	1.050 × 10 ⁻⁴	9.624 × 10 ⁻⁵

Correlation for	Deviation from experimental data	R square	Standard error
Relative pH	-2.01% to 1.89%	0.967	0.0107
Relative electrical conductivity	-8.11% to 8.95%	0.954	0.0674
Relative viscosity	-6.72% to 9.33%	0.921	0.0553

TABLE 2 Measure of deviation between the correlated data and experimental values.

development of ions or charges at room temperature. However, the increase in temperature might dissociate the fluid molecules and particle constituents into charged ions. As discussed in the previous section, while the significant component of the bio powder, CaO, is alkaline, they completely dissociate into charged particles besides the dissociation of glycols to generate OH⁻ to increase the total number of ionic charges in the liquid. The phytochemical constituent that contributes to lower pH also produces charges that can increase thermal conductivity. The sudden rise in the electrical and relative electrical conductivity beyond 40°C is due to the same explanation provided previously. Moreover, this sudden upsurge of values happens when the volume concentration increases beyond 0.4%.

3.5 | Effect of nanoparticles concentration and temperature on the viscosity of nanofluid

Viscosity is one of the most critical thermo-physical parameters that determine the suitability of the fluid for specific applications particularly for flowing situation. Besides temperature, the nanofluid's

viscosity mainly depends upon the base fluid's viscosity, particles loading, size, and shape. As can be noticed from Figure 9, the increase in viscosity as a function of particle loading is due to the excess interaction between the particle and fluid molecules. However, like any liquid the viscosity of the nanofluid decreases when prone to a higher temperature. It attributes to breaking intermolecular forces prevailing between the solid-liquid interfacial layers, prompting the Brownian mechanism.

The decrease in viscosity at elevated temperatures attributes to the dissociation of CaO. It is a general characteristic of CaO that it fully dissociates at high temperatures, thus being less viscous. In addition, the phytochemicals in the tree bark powder aid in the dissolution of the powder into the base fluid at a higher temperature, thus reducing the viscous behavior of the liquid. As keenly observed from Figure 7, there is a steep rise in the relative Viscosity of the highly concentrated nanofluid; especially from 0.6% concentration is notable. The reason for this inference must be that after a particular volume concentration, the phytochemicals might be saturated and not dissolve further within the base fluid, generating shear behavior. The maximum viscosity was 27.91 mPa s at 20°C for 1.0% concentrated

nanofluid, while the lowest value was 3.9 mPa s at 70°C for 0.2% concentration. Thus, FTB-EG gave minimal viscosity enhancement that can contribute to reduce clogging and lower pressure drop.

3.6 | Development of correlation

The correlations for the measured properties as a function of temperature and concentration were developed based on the available data. The common correlation applies to the entire range of temperature and volume concentrations. The correlation for relative (nanofluids/base fluid) properties is expressed in the below form

$$R_p = A_1 + (A_2\phi) + (A_3T) + (A_4\phi T) + (A_5\phi^2) + (A_6T^2) + (A_7\phi^2T^2), \quad (1)$$

where, R_p represents relative properties (viscosity, electrical conductivity, and pH), A_1 – A_7 are correlation (empirical) constants, T is the temperature, and ϕ is the volume fraction of FTB nanoparticle.

Table 1 provides the values for the constants A_1 to A_7 for the corresponding the properties, and Table 2 provides the quantity of deviation from the experimental data.

4 | CONCLUSIONS

The physical properties such as viscosity, pH, and electrical conductivity of FTB-EG natural (bio) nanofluids have been investigated under varied temperatures and volumetric concentrations of FTB nanoparticles. The pH, viscosity, and electrical conductivity augmented with a rise in concentration. However, while the electrical conductivity enhanced with a temperature rise, the other two properties behaved exactly the opposite. The findings of this study are summarized as follows:

- i. The morphological study confirmed that the FTB particles are of spherical shape and have the average particle size is 26 nm.
- ii. Oxides of calcium and silicon were the two major constituents of this bioproduct.
- iii. The highest viscosity enhancement is 75% at 1.0% concentration and at 70°C, while the lowest is 1.02 at 0.2% and at 20°C.
- iv. The maximum pH enhancement is 26% at 1.0% concentration at 40°C, while the minimum is 9% at 0.2% concentration and at 20°C.
- v. The superior electrical conductivity enhancement is 104% at 1.0% concentration and at 70°C, while the inferior is 2% at 0.2% concentration and at 20°C.

This study will contribute toward the exploration and application of such environmentally friendly natural nanofluids. However, more studies are to be conducted on various areas of this nanofluid.

AUTHOR CONTRIBUTIONS

Suseel Jai Krishnan: Validation; writing – review and editing; data curation. **J. T. Awua:** Investigation; writing – original draft. **J. S. Ibrahim:** Supervision; formal analysis; conceptualization. **A. O. Edeoja:** Methodology; formal analysis. **A. Kuhe:** Conceptualization; investigation. **M. Sharifpur:** Formal analysis; supervision; project administration; resources. **S. M. S. Murshed:** Funding acquisition; visualization; validation.

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CONFLICT OF INTEREST STATEMENT

There is no potential conflict of interest.

DATA AVAILABILITY STATEMENT

Research data are not shared.

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