

Ionic Conductivity Enhancement in LiClO₄-Doped JSS-PVA Biopolymer Electrolytes for Sustainable Energy Storage

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ABSTRACT

Biopolymer electrolytes are gaining attention due to their eco-friendly, biodegradable nature, offering a sustainable alternative to conventional, fossil fuel-derived polymer electrolytes. Traditional polymer electrolytes often rely on non-biodegradable materials and use liquid components prone to leakage, reducing device reliability. Although solid polymer electrolytes eliminate leakage issues, their low ionic conductivity remains a major limitation for practical applications. This study aims to develop an environmentally friendly solid polymer electrolyte by blending jackfruit seed starch (JSS) with polyvinyl alcohol (PVA), incorporating varying concentrations of lithium perchlorate (LiClO₄) from 10 to 50 wt.% using the solution casting technique. The goal is to optimize the blend for maximum ionic conductivity suitable for electrochemical devices. Impedance spectroscopy revealed that the highest ionic conductivity of 0.0025 S/cm occurred at 40 wt.% LiClO₄. Dielectric permittivity (ϵ') peaked at low frequencies due to electrode polarization, also at 40 wt.%. Electric modulus (M') analysis indicated enhanced charge carrier mobility, while transference number measurements showed that ionic contribution accounted for 87% of conductivity. Linear sweep voltammetry confirmed good electrochemical stability between 3.0 and 4.5 V. These findings demonstrate the potential of LiClO₄-doped JSS-PVA blends as sustainable, high-performance solid electrolytes for next-generation energy storage systems.

Keywords: conductivity; jackfruit seed starch; lithium perchlorate; polyvinyl alcohol

INTRODUCTION

Polymer electrolytes (PEs) are materials composed of linear macromolecular chains capable of conducting ions, making them essential components in a wide range of

electrochemical devices such as batteries, supercapacitors, and solar cells. Their role in enabling efficient energy storage and conversion has garnered significant attention, particularly as the demand for sustainable and high-performance materials continues to grow. Most conventional polymer electrolytes come from petroleum-based, non-biodegradable polymers. These materials cause environmental pollution, have high production costs, and depend on limited fossil fuel resources [1].

The development of eco-friendly and biodegradable substitutes made from natural polymers is gaining popularity in response to these challenges. Jackfruit seed starch (JSS) is one of these options. JSS is a biodegradable and renewable biopolymer that contains high levels of amylose and amylopectin. These components create channels for ionic conduction and provide flexibility. Additionally, starch's ability to attract water helps improve ion mobility and dissociation. However, JSS's poor mechanical strength limits its use in high-performance situations. To address this problem, JSS is mixed with polyvinyl alcohol (PVA), a water-soluble, biodegradable polymer known for its strong film-forming and mechanical properties. The hydrogen bonding between the hydroxyl groups in PVA and the starch enhances the blend's compatibility, elasticity, and stability. PVA also boosts thermal and chemical stability, which makes the composite more durable in real-world applications [2].

A number of benefits arise from combining JSS and PVA, such as better ionic conductivity, homogenous structure, and mechanical characteristics. According to Ramli et al. (2022), both materials are hydrophilic, which promotes ion transport, particularly when doped with appropriate salts like lithium perchlorate (LiClO_4) [1]. Salt doping improves the electrical and electrochemical characteristics of a solid polymer electrolyte by introducing mobile ions into the polymer matrix. Nevertheless, solid polymer electrolytes usually have low ionic conductivity, which limits their practical use even with these improvements.

This study aims to optimize a JSS-PVA-based solid polymer electrolyte system with various LiClO_4 concentrations. The goal is to develop optimum ionic conductivity and electrochemical stability, lead the system ideal for practical energy storage applications including batteries, supercapacitors, and fuel cells. This technique is consistent with green chemistry principles and promotes the development of sustainable materials for next-generation electrochemical devices.

MATERIALS AND METHOD

The JSS-PVA polymer blend electrolyte was synthesized using a solution casting method. The jackfruit seeds were first collected, cleaned of surface contaminants, and peeled to extract the inner seeds. The samples were dried in an oven at 60 °C for 24 h to remove moisture. The dried seeds were ground into a fine powder with a mechanical grinder before being sieved through a 100-mesh screen to ensure uniform particle size. The powdered jackfruit seed starch (JSS) was then heated to 80 °C after being mixed with distilled water in a 1:20 (w/v) ratio. A clear viscous solution was formed after 30 min of constant stirring with a magnetic stirrer, followed by the addition of 2 mL of glycerol, 1 mL of NaOH, and 1 mL of HCl. The mixture was then allowed to cool to room temperature. In parallel, the PVA solution was prepared by dissolving PVA powder in distilled water at a 1:20 (w/v) ratio, heated to 90 °C with constant stirring until fully

dissolved, and subsequently cooled. The JSS and PVA solutions were blended in a 1:1 ratio under continuous stirring for 30 min to form a homogeneous mixture, which was then left undisturbed for 10 min to remove air bubbles. Lithium perchlorate (LiClO_4) was added to the blend at concentrations ranging from 10 to 50 wt.%, followed by stirring for 1 h at room temperature to ensure even salt distribution. The final mixture was cast into clean glass petri dishes to form thin films, which were left to dry at room temperature for 48 h. The dried films were then carefully peeled off and stored in a desiccator to prevent moisture absorption. Then, the sample were characterized by using an Electrochemical impedance spectroscopy (EIS) to determine the ionic conductivity of the polymer electrolyte films using stainless steel blocking electrodes over a frequency range of 0.1 Hz to 1 MHz at room temperature. The bulk resistance (R_β) was obtained from Nyquist plots, and ionic conductivity (σ) was calculated using

$$\sigma = t/(R_\beta A) \quad (1)$$

where t is the film thickness and A is the electrode–electrolyte contact area [3]. The electrochemical stability window was evaluated by linear sweep voltammetry at a scan rate of 10 mV s^{-1} using a stainless steel/electrolyte/stainless steel configuration. The ionic transference number was determined by DC polarization at 1 V from the initial and steady-state current response.

RESULTS AND DISCUSSION

Impedance and conductivity analysis

Figure 1(a) shows the Nyquist plot for the electrical response of PVA–JSS polymer electrolyte films with varying concentrations of LiClO_4 (10–50 wt.%). In typical electrochemical impedance spectra of polymer electrolytes, a semicircle in the high-frequency region is often attributed to the bulk resistance and capacitance of the material. In the present study, distinct semicircular to bulk resistance were not observed in the Nyquist plots for all samples. Instead, the impedance spectra are dominated by a low-frequency spike with varying inclination, indicating the presence of blocking electrode effects and highly capacitive behavior at the electrode–electrolyte interface. Such spike-dominated spectra are commonly reported for polymer electrolytes with high ionic conductivity, where the bulk resistance is too small to be resolved within the measured frequency range [4]. The inclined spike observed for the sample containing 10 wt.% LiClO_4 suggests ion transport governed by a combination of ionic diffusion and interfacial polarization rather than pure Warburg diffusion [5]. Therefore, the ionic conductivity was determined from the intercept of the spike with the real axis. As the concentration of LiClO_4 increases, the angle of this spike gradually becomes lower (more vertical), signalling a transition from diffusion-limited to more capacitive or bulk-dominated ionic conduction [3],[6]. This behavior can be explained by the increased availability of free lithium ions at higher salt concentrations, which facilitates faster ion transport and reduces the overall impedance. The decreasing angle of the spike with increasing salt concentration indicates improved ion diffusion and reduced resistance at the electrode–electrolyte interface, supporting the conclusion that higher LiClO_4 content enhances the electrochemical performance of the system. As the concentration of LiClO_4 increases up

to 40 wt.%, the intercept of the impedance curve on the real axis (Z') decreases, indicating a reduction in bulk resistance. Hence, this suggests that ionic conductivity improves with increasing salt content due to the higher number of mobile charge carriers (Li^+ ions) available for transport. At 40 wt.% LiClO_4 , the system exhibits the R_b value showed the lowest value, corresponding to the highest ionic conductivity among all samples. However, at 50 wt.%, the impedance increases again, suggesting a decline in conductivity.

Figure 2 illustrates the variation in ionic conductivity of PVA–JSS blend biopolymer electrolytes (BPEs) with increasing concentrations of LiClO_4 from 10 wt.% to 50 wt.%. The trend reveals a significant dependence of conductivity on salt concentration, aligning well with the impedance analysis shown in Figure 1. As the concentration of LiClO_4 increases from 10 wt.% to 40 wt.%, the ionic conductivity rises steadily, reaching a maximum value of 0.0025 S/cm at 40 wt.%. The availability of Li^+ ions enhance ionic conduction in the polymer electrolyte, leading to a low bulk resistance. As a result, no well-defined semicircle is observed in the high-frequency region of the Nyquist plots, which is characteristic of highly conductive polymer electrolytes.

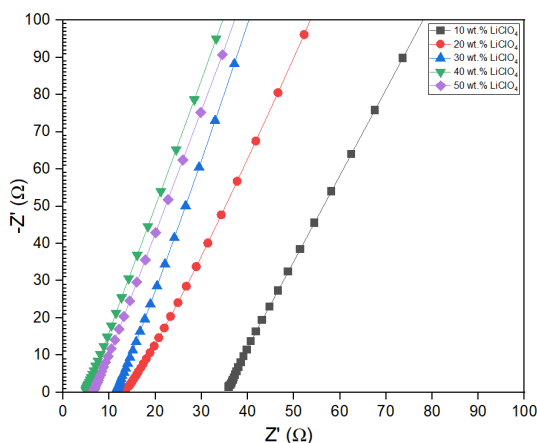


Figure 1. Nyquist plot with different concentration of LiClO_4

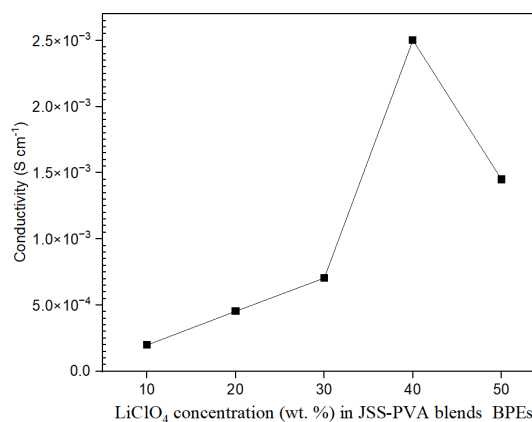


Figure 2. Conductivity of different concentration of LiClO_4 in CS-PVA blend SPEs

The hydrophilic nature of both JSS and PVA facilitates improved ion mobility by promoting better salt dissociation and transport pathways. However, at 50 wt.%, a decline in conductivity is observed. This drop is likely due to ion aggregation or ion pairing, where excess Li^+ and ClO_4^- ions begin to cluster, reducing the number of free mobile charge carriers and thus hindering ionic motion. Excessive salt content may lead to polymer chain stiffening or even phase separation, which compromises the homogeneity of the electrolyte matrix [7]. This phenomenon is comparable to earlier findings on PEO, where the incorporation of LiClO_4 was observed to increase the rigidity of the polymer chains, thereby reducing the flexibility of the matrix. This behaviour indicates an optimal salt concentration (~ 40 wt.%) where the balance between free-ion availability and polymer segmental motion maximizes conductivity. According to equation (2), the conductivity of ions also depends on the concentration of charge carriers. The polymer

host dissolves all Li^+ when the concentration of salt is sufficiently low, allowing all Li^+ to be utilized as charge carriers. In line with

$$\sigma = \sum n_i q_i \mu_i \quad (2)$$

too little salt limits the carrier concentration n_i , whereas excess salt promotes ion pairing and hinders segmental dynamics, reducing μ_i so σ falls beyond the optimum [8]. Nevertheless, the electrostatic interaction between cations and anions becomes non-negligible as concentration rises, potentially lowering the number of charge carriers. Hence, beyond this concentration, the system becomes oversaturated, leading to reduced ionic efficiency. This finding is consistent with previous work on Polyvinyl alcohol-Chitosan (PVA-CS) blends: at Sodium Perchlorate (NaClO_4) loadings above ~35 wt.%, the conductivity decreases due to ion re-association, whereas at lower loadings the limited number of dissociated ionic carriers restricts conductivity [5].

Dielectric permittivity and Modulus analysis

The variation of the dielectric permittivity with frequency was studied in the variation of the concentration wt.% of LiClO_4 in the JSS-PVA blend at room temperature, as shown in Figure 3(a). Dielectric analysis revealed that the dielectric permittivity (ϵ') exhibited high values at lower frequencies, primarily due to electrode polarization effects [9]. At these frequencies, ions tend to accumulate at the electrode-electrolyte interface, forming a double-layer capacitance that significantly enhances the material's charge storage capacity [10,11]. It should be noted that this high dielectric permittivity at low frequencies is consistent with the spike-dominated Nyquist plots observed in impedance analysis, where strong electrode polarization suppresses the bulk semicircle while enhancing ϵ' due to interfacial charge accumulation. This interfacial polarization is particularly beneficial for polymer electrolytes, as it improves their ability to store and transfer charge at reduced frequencies. As the frequency increased, a sharp decline in dielectric permittivity was observed. This behavior is attributed to the inability of dipoles within the polymer matrix to keep pace with the rapidly oscillating electric field, resulting in dielectric relaxation [11]. Such relaxation behavior is consistent with the findings of Kumbhakar et al. (2023), who reported that dielectric properties are strongly influenced by both ionic mobility and dipolar response dynamics.

The ϵ' values increased with the increment of salt concentration in the polymer blend electrolytes. The highest dielectric permittivity was recorded at 40 wt.% LiClO_4 , where a favorable balance between the number of free charge carriers and the amorphous content of the polymer matrix facilitated efficient polarization. Hence, this figure exposes the higher conducting electrolyte of the JSS-PVA polymer blend electrolyte, which achieved a higher value of the dielectric constant because more charge accumulation occurs in the high-frequency region. This finding agrees with the previous reports in which dielectric permittivity increased with the concentration of guanidinium carbonate (GuC) up to 25 wt.% in a poly(vinylpyrrolidone)-poly(ethylene oxide) (PVP/PEO) blend [11]. At high salt concentrations, the formation of ion pairs and a large number of aggregates was found to act as a plasticizer, thereby facilitating ion transport through the electrolyte. However, when the LiClO_4 salt concentration becomes excessive, which is 50 wt.%, ion aggregation dominates, restricting ion mobility and ultimately reducing

overall permittivity. Overall, these results underscore the significance of the 40 wt.% LiClO₄ composition in the JSS-PVA system as an optimal balance, offering enhanced charge storage and dielectric response due to its effective polarization behavior.

The frequency dependence of the real part of the electric modulus (M') for JSS-PVA polyblend samples at room temperature is presented in Figure 3(b). Electric modulus analysis provides valuable insight into the relaxation behavior and stiffness of PVA–JSS-based polymer electrolytes doped with varying concentrations of lithium perchlorate (LiClO₄). At low frequencies, M' remains close to zero due to the dominant effect of electrode polarization and the limited contribution from the bulk material, reflecting slow ionic motion and interfacial charge accumulation [12]. As frequency increases, M' increases nonlinearly and eventually approaches saturation, indicating a transition toward bulk-dominated response as interfacial polarization effects diminish [13]. The results also show that salt concentration plays a significant role in modulating the modulus response. The sample with 10 wt.% LiClO₄ displays the highest M' values, while the 40 wt.% sample exhibits the lowest, indicating differences in short-range charge carrier mobility within the LiClO₄–JSS-PVA electrolyte matrix [13]. The divergence of the modulus curves at higher frequencies further confirms the strong correlation between ionic content, relaxation dynamics, and the frequency-dependent stiffness of the polymer electrolyte.

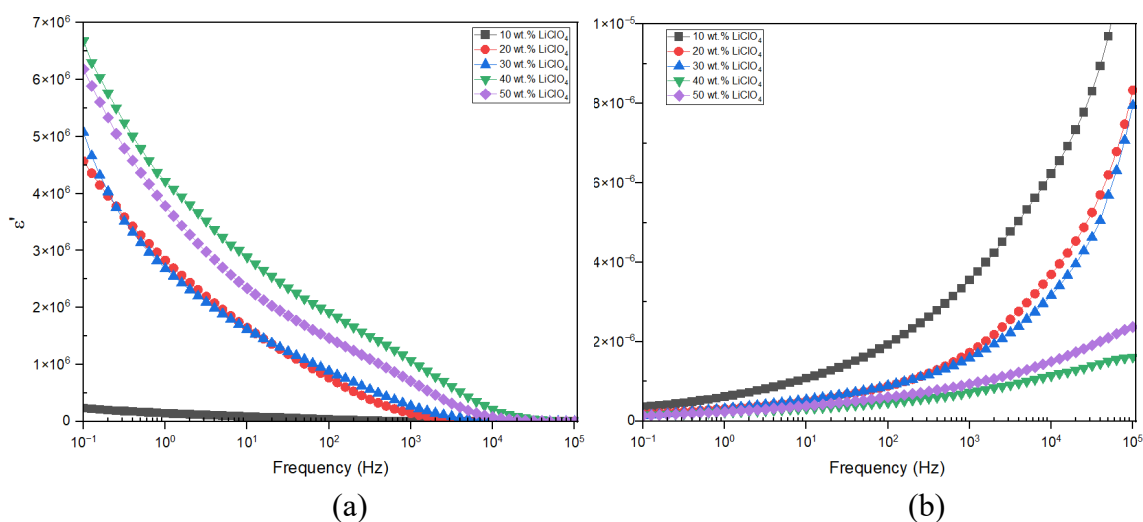


Figure 3. Variations in (a) dielectric (ϵ') and (b) Modulus (M') across different concentration of LiClO₄ in JSS-PVA blend polymer electrolyte

Linear Sweep Voltammetry (LSV) analysis

Linear sweep voltammetry (LSV) is a technique for quantitatively describing the voltage stability range (ESW) of SPEs. A larger ESW not only broadens the application scope of electrolytes but also allows for the development of high-voltage, high-energy density energy storage devices [14]. The LSV measurement was carried out using 5 mV.s⁻¹ scan rate. LSV plot for all conducting samples is displayed in Figure 4. The Linear Sweep Voltammetry (LSV) analysis was conducted to evaluate the electrochemical stability of JSS–PVA polymer electrolytes doped with varying concentrations of lithium perchlorate (LiClO₄), ranging from 10 to 50 wt.%. As shown in the LSV plots, all samples exhibit negligible current in the voltage range of 0 to 3 V, indicating minimal electrochemical

activity and the absence of significant redox reactions within this region. This behavior suggests good electrochemical stability under moderate voltages. The voltage range in which a significant increase in current occurs indicates an unsafe voltage region during cell operation[14]. A sharp increase in current is observed around 3.0 V for all compositions, marking the onset of electrochemical decomposition or oxidative breakdown of the electrolyte [15]. The onset potential and current intensity are influenced by the LiClO_4 concentration, reflecting a direct relationship between ionic conductivity and electrochemical performance. The observed trend reveals that polymer electrolytes with low ionic conductivity exhibit higher oxidative potential in LSV analysis. This is because, at low salt concentrations, most anions (ClO_4^-) remain strongly coordinated with Li^+ ions and the polymer backbone, leaving very few free anions available for electrochemical oxidation at the electrode surface. As a result, the system demonstrates greater electrochemical stability, reflected in a higher oxidative potential. Conversely, as the salt concentration increases, a larger fraction of free anions and ion aggregates are present. These species are more susceptible to oxidation, thereby reducing the stability window despite the enhanced ionic conductivity arising from the higher density of mobile charge carriers. This trade-off highlights the inverse relationship between conductivity and oxidative stability, where electrolytes with low conductivity maintain high stability, while those with high conductivity exhibit reduced oxidative potential. Nevertheless, all samples investigated in this study demonstrate sufficient stability due to the oxidative value obtained more than 1V to be applicable for energy storage [16].

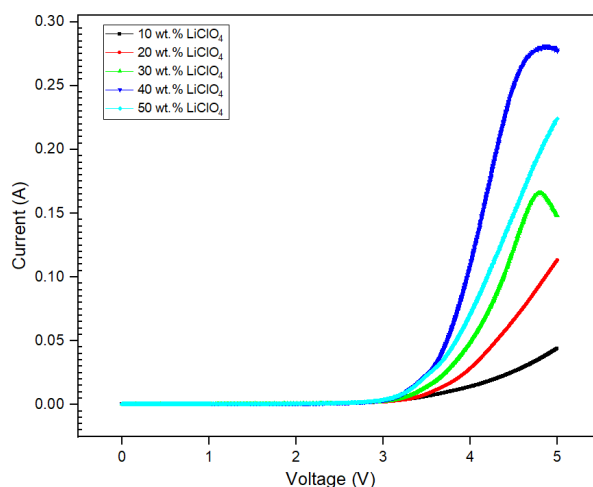


Figure 4. LSV curves with decomposition voltage of the samples with variation concentration of LiClO_4

Transfer Number Measurement analysis

In this method used to support the conductivity measurement and validate the ionic species of the JSS–PVA blend polymer electrolyte system by D.C Wagner Polarization method. This process is mainly contributed only by the ions and negligible amounts of electrons[17]. Figure 5 shows the polarization plot as a function of time versus current at room temperature for the best conductivity system. The transference number

measurement (TNM) provides key insights into the charge transport mechanisms within the JSS–PVA–LiClO₄ polymer electrolyte system [18]. The TNM plot reveals a two-phase behavior: an initial sharp current associated with electronic charge carriers, followed by a gradual decline to a steady-state plateau dominated by ionic transport. The rapid reduction of electronic current indicates that electrons contribute only transiently to the overall conductivity, while the stable plateau confirms the sustained role of ions in charge transport. By using Equations (3) and (4), the percentage of the total ionic transference number (t_{ion}) was calculated from the initial (I_i) and final (I_f) currents according to

$$t_{ion} (\%) = \left(\frac{I_i - I_f}{I_i} \right) \times 100 \quad (3)$$

while the electronic transference number (t_e) was obtained using

$$t_e (\%) = 100 \% - t_{ion} \% \quad (4)$$

Quantitative analysis reveals that ionic conductivity contributes 84.62 % of the total conductivity, whereas electronic conductivity accounts for only 15.38%, confirming the predominantly ionic nature of charge transport in the JSS–PVA–LiClO₄ polymer electrolyte. This significant dominance of electronic transport highlights a current limitation of the system for applications requiring efficient ionic conduction, such as batteries and supercapacitors. These findings are in line with observations by Aziz et al. (2024), who reported similar dual-mode transport behavior in chitosan- polyvinyl alcohol- sodium acetate biopolymer-based electrolytes.

Despite the relatively low ionic contribution, the presence of stable ionic transport is promising. It establishes a foundation for further optimization, particularly through structural or compositional modifications aimed at enhancing the ionic transference number. Improving ion mobility and suppressing electronic leakage will be essential to realize the full potential of this biopolymer-based electrolyte in next-generation electrochemical energy storage devices.

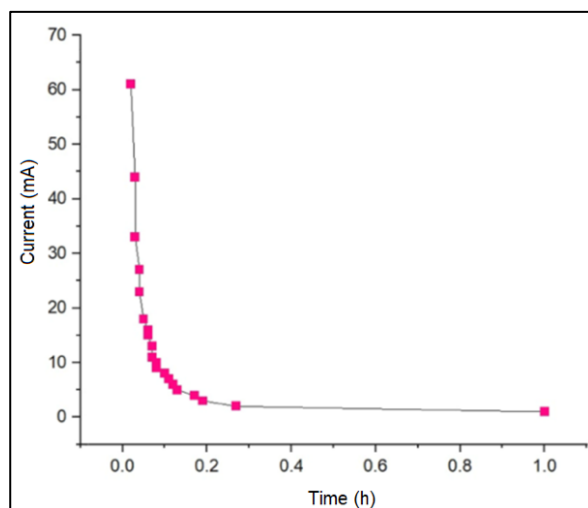


Figure 5. The TNM plot for the optimal conducting polymer electrolyte

CONCLUSIONS

A biodegradable solid polymer electrolyte (SPE) based on a jackfruit seed starch (JSS)–polyvinyl alcohol (PVA) blend with lithium perchlorate (LiClO₄) was successfully developed and characterized. The 40 wt.% LiClO₄ composition exhibited the highest ionic conductivity (0.0025 S/cm), favorable dielectric response, and stable electrochemical performance up to ~3.0 V, reflecting an optimal balance between ion availability and polymer chain mobility. The transference number analysis showed that ionic conduction contributed only 84.62% of the total conductivity, the results highlight the potential of JSS–PVA–LiClO₄ composites as sustainable polymer electrolytes. Future work should focus on enhancing ionic transport and reducing electronic leakage, for example through structural modification or nanofiller incorporation, to further improve their suitability for advanced energy storage applications.

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AUTHOR'S CONTRIBUTION STATEMENT

Conceptualization, F.A.L.; Methodology, N.H.R.M.N.A., A.I.A.G. and M.D.M.M.F.; Analysis and investigation, N.I.A.M.O., M.A.A.A., and N.A.M.S.S.; Validation, F.A.L.; Data curation, F.A.L.; Writing – Original Draft Preparation, N.B.M.; Writing – Review & Editing, N.I.A.M.O. and F.A.L.; Supervision, F.A.L.

CONFLICT OF INTEREST

All authors declare no conflict of interest.

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