



Research Papers

Card-bisphenol-based hybrid polybenzoxazine composites for high-*k* dielectrics: Activated vs non-activated *Phragmites karka* carbonJanapriya Mathimani^a, Sasikumar Ramachandran^{a,*}, Poornesh Devaraj^a,
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ABSTRACT

Bio-based benzoxazine (CBPA-Bz) resin was synthesized using card-bisphenol derived from cardanol, and aniline with paraformaldehyde through Mannich condensation followed by thermal polymerization to obtain polybenzoxazine (CBPA-PBz). Free hydroxyl groups of the polybenzoxazine matrix developed on polymerization have been modified in the form of ester via esterification with dodecenyl succinic anhydride (DSA) at high temperature through in situ reaction. The non-activated bio-carbon (C_{na}) and acid-activated bio-carbon (C_{aa}) were derived from *Phragmites karka* (*p.karka*). Different weight percentages of bio-carbons reinforced hybrid CBPA-DSPA-PBz- $C_{na/aa}$ polybenzoxazine composites were prepared, and studied their dielectric, thermal, and water-repellent properties. FTIR and ¹H NMR spectra were used to ascertain the molecular structure of the benzoxazine monomer and polymer composites. The hybrid CBPA-DSPA-PBz and its composites exhibit higher values of water contact angle (WCA) between 141° and 165° infer their (super) hydrophobic behaviour compared to its pristine CBPA-PBz matrix (WCA = 140°). The hydrophobic property of the developed polybenzoxazine (CBPA-DSPA-PBz) and its composites (CBPA-DSPA-PBz- $C_{na/aa}$ 1-10) could provide an authentic dielectric constant and dielectric loss values, because of preventing the effect of moisture on dielectric measurement. The CBPA-DSPA-PBz- C_{na} 10 and CBPA-DSPA-PBz- C_{aa} 10 hybrid composites show high values of dielectric constant, 6.97 and 7.55, respectively.

1. Introduction

Dielectric materials are insulators and are used in the electrical and electronic applications, they are essential for controlling the effect of electric fields. They are categorized into low-*k* and high-*k* dielectrics based on their values of dielectric constant (relative permittivity, ϵ_r), which discloses their ability to store electrical energy in an electric field. High-*k* dielectric materials have a high value of dielectric constant relative to silicon dioxide dielectric constant value of 3.94, and are used in semiconductor devices to increase speed, reduce power consumption, and improve performance of the devices. Therefore, they are essential in power electronics applications such as miniaturization of transistors, increase capacitance in capacitors, and reduce leakage currents, particularly in microelectronics [1–4].

The conventional polymer matrices limit their applications required for high-performance of electronic devices [5,6]. To overcome this

drawback, Polybenzoxazines (PBzs) based composites possessing high-*k* dielectrics can be a choice, as they generally exhibit greater electrical and thermal properties than those of conventional polymers [7,8]. These consequences were replaced with PBzs due to their exceptional dimensional stability, high thermal stability, good flame-retardant behaviour, low-moisture absorption, excellent adhesive strength, catalyst-free polymerization, lightweight, good mechanical properties, high glass transition temperature (*T_g*), low surface free energy and good electrical properties. Additionally, PBzs offer smart macromolecular design flexibility, near-zero volumetric expansion, high char yield, and rapid improvement in physical and mechanical properties at low conversion. These advantages make PBzs an appealing alternative to traditional thermosetting resins such as phenolic, polyimide, epoxy, and bismaleimides [9–11].

The use of natural phenolic and polyphenolic compounds, including cardanol, vanillin, eugenol, urushiol, lignin, gallic acid, and coumarin,

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500 °C to yield 12 % char at 850 °C. Whereas, the hybrid CBPA-DSA-PBz exhibits 29 % weight loss in the first degradation between 230 °C and 370 °C which might be due to the cleavage of ester linkages including the long aliphatic side chain of the cardanol unit, following the decomposition of polybenzoxazine networks occurred between 390 °C and 500 °C and 8 % char yield was obtained at 850 °C. Similar trend was observed in the case of bio-carbon reinforced CBPA-DSA-PBz- $C_{na/aa}$ 1-10 hybrid composites (Fig. 11), however, the char yield was increased up to 25 wt. % (CBPA-DSA-PBz- C_{na} 10) with increasing the C_{na} content from 1 wt. % to 10 wt. % and the results obtained are presented in Table 2. The increased char yield considerably varied between C_{na} and C_{aa} reinforced polybenzoxazine composites, the activated carbon reinforced hybrid CBPA-DSA-PBz- C_{aa} 10 composites exhibit 19 % char yield at 850 °C. This might be due to the presence of hydroxyl, carboxyl and carbonyl functional groups may interact with the polymer matrix and leads to degrade along with them, which influences the thermal decomposition and contributes to lower char yield at 850 °C.

4.7. Water contact angle of the polybenzoxazine composites

The water repellent/attraction behaviours of the materials could affect the dielectric properties of that materials because of the dielectric constant of water is about 80 at room temperature. Hence, it is desirable to study the water contact angle of the developed polybenzoxazine composites. As shown in Fig. 12, the hybrid CBPA-DSA-PBz (158°) exhibits the higher value of water contact angle namely super hydrophobic when compared to that of neat CBPA-PBz (140°) which infers the presence of additional long aliphatic chain in the cross-linker and the formation of ester linkages significantly influenced to enhance the value of water contact angle. Further, the C_{na} reinforced hybrid CBPA-DSA-PBz- C_{na} 1-10 composites exhibit higher water contact angle in the ranges of 158-165° which infers their super hydrophobic property. Whereas, the C_{aa} reinforced hybrid CBPA-DSA-PBz- C_{aa} 1-10 composites displays the decreasing trend of the value of water contact angle with increasing percentage of C_{aa} which might be due to the presence of polar functional groups, intermolecular hydrogen bonding and modifying the surface free energy of the composites [68].

5. Conclusion

Utilisation of agricultural waste like cardanol derived from cashew nut shell liquid, and *p. karka* plant-based carbon for the development of polymer composites is an effective approach to obtain bio-based dielectrics. In the present work, a sustainable bio-based polybenzoxazine composites (CBPA-DSA-PBz- $C_{na/aa}$ 1-10) were developed as high- k dielectrics using card-bisphenol-based benzoxazine (CBPA-Bz), an anhydride (DSA), and non-activated (C_{na})/acid-activated (C_{aa}) bio-carbon. The neat CBPA-PBz matrix inherently exhibits the higher value of dielectric constant of 5.55 with hydrophobic nature, while the hybrid polybenzoxazine (CBPA-DSA-PBz) shows the lower value of dielectric constant (3.39) with super-hydrophobic behaviour. Further, the non-activated (C_{na}) or activated biocarbon (C_{aa}) reinforced hybrid polybenzoxazine composites exhibit an increased value of dielectric constant with increasing weight percentages of bio-carbon. The influential effect of acid-activated bio-carbon could increase the value of dielectric constant ($k = 7.55$) of the hybrid CBPA-DSA-PBz- C_{aa} 10 when compared to that of non-activated bio-carbon reinforced hybrid CBPA-DSA-PBz- C_{na} 10 composite ($k = 6.97$). This might be due to the oxidative products of polar functional groups, such as hydroxyl, carbonyl, and carboxylic acid present in the acid-activated carbon. Further, the developed polybenzoxazine composites remain thermally stable up to about 230 °C. After that, the composites underwent a two-step thermal degradation due to the decomposition of ester-linkages and long aliphatic side chain, following the polybenzoxazine networks. The present work provides an avenue for the development of bio-based high- k dielectrics and can create an impact in the miniaturization and advancement of the modern

electronics devices.

Ethics approval and consent to participate

Not applicable.

Consent for publication

Not applicable.

CRediT authorship contribution statement

Janapriya Mathimani: Writing – original draft, Data curation. **Sasikumar Ramachandran:** Writing – review & editing, Supervision, Methodology, Investigation, Conceptualization. **Poornesh Devaraj:** Data curation. **Dharani Shri Ramachandran:** Formal analysis, Data curation. **Alagar Muthukaruppan:** Writing – review & editing, Project administration, Funding acquisition.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Data availability

Data will be made available on request.

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