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Improvising of flame, thermal and microbial degradation stability of waste cotton microfibre vinyl ester composites using annealed-silane modified barely husk biosilica

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The present study examines how waste cotton microfiber-vinyl ester composites' flammability, thermal stability, and antibacterial qualities are affected by annealed-silane modified biosilica. Specimens were carefully manufactured using the solution casting method and thoroughly characterized in accordance with ASTM guidelines. Positive results from the flammability evaluation showed that adding annealed-silane modified biosilica could improve fire resistance. Notably, the VCB3 composite achieved a V-0 rating by showing decreased flammability, enhanced fire retardancy, and a decreased velocity of propagation or propagation speed of 9.77 mm/min. Moreover, it exhibited no cotton lightens and falling drops. These results hold significant promise for applications where fire safety is of utmost importance. In terms of thermal stability, the modified composites displayed heightened resistance to elevated temperatures. Specifically, the VCB3 composite showcased remarkable thermal stability, featuring a notably high initial decomposition temperature of 388°C.Furthermore, the investigation into the antimicrobial properties of these composites unveiled their potential as materials inherently resistant to microbial growth. Similar to the improvements seen in flammability and thermal stability, VCB3 demonstrated enhanced antimicrobial characteristics, with increased inhibitory diameters of 14.36mm and 14.72mm against S. Aureus and E. Coli, respectively. This quality holds substantial promise for various industries, particularly those emphasizing hygiene and microbial control.These results present promising opportunities for the use of these advanced composites in a variety of fields where fire safety, resilience to high temperatures, and antibacterial properties are desirable, such as healthcare, transportation, and construction.

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Keywords: Composites, Biofibre, Silica, Flammability, Microbial activities

1. Introduction

Utilization of materials has evolved alongside humankind, from the Stone Age to the current day, and will likely continue to do so. This breakthrough in materials research has implications for composites as well. Composites are typically made up of two or more compounds that each have their own unique physical, chemical, and mechanical characteristics [1]. Composites can be further categorized by the types of materials used to create the matrix, which include metal, ceramic, and polymer [2]. Lightweight, excellent resistance to wear and corrosion, and high stiffness and strength are only some of the reasons why polymer matrix composites have attracted so much attention [3–4]. Polymer matrix composites are used in a wide variety of industries due to their advantageous characteristics and properties [5, 6]. Some examples of these

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include the automotive, aircraft and aerospace industry, marine, sports goods, biomedical, electrical and electronic equipment's, and various protective equipments in the defense military industry. In addition to these uses, polymer matrices composites are made up of a wide range of short or continuous fibers held together by an organic polymer matrix. The polymer matrix is then supplemented with natural fiber and natural filler material [6] to create a biodegradable and sustainable eco-friendly biocomposite material. As a result, heat degradation can cause the polymer chain to break and react with itself, causing the polymer's characteristics to alter (e.g., becoming less ductile, more brittle, cracking, changing color, etc.) [7].

Some examples of natural fibers that could be utilized to increase polymer composites' resistance to degradation include kenaf, hemp, sisal, cotton, jute, flax, sunflower, abaca, areca, aleovera, and silk. Among various fiber materials, the cotton fiber is utilized since prehistoric period and it is cultivated widely in tropical and subtropical regions of the world. India is the greatest cotton producer in the world, with a share of about 15% of the global total [8]. The cellulose concentration of this cotton fiber is above 90%, and it may also contain trace amounts of waxes, lipids, pectin, and water. The factory-level processing of this massively farmed and cellulose-rich cotton plant has resulted in substantial losses. An environmentally benign and useful option for recycling textile mill scraps is to use them as fiber reinforcement in composite material production. This could be another option for reducing the amount of trash dumped on Earth every year. In addition to this, for further increase the qualities of the cotton fiber it might be treated using silanes to eliminate the surplus cellulose and incept adhesion linkages for greater adhesion and compatibility with resin.

In addition to its well-documented benefits (low density, good heat conductivity, high flexibility, resilience, elasticity, and renewability), the tensile strength of cotton fiber material is known to be increased due to its high cellulose content [9]. Unsaturated polyester-based composites bonded with 100% cotton fabric waste material for fake ceiling board application have been the subject of research by Gedif et al [10]. The author discovered that increasing the percentage of cotton fabric to 33 wt.% in an unsaturated polyester composite increased the composite's maximum tensile strength from 198 MPa to 1105.3 MPa, and increased its flexural strength from 30.1 MPa to 1105.3 MPa. The effects of cotton fiber surface treatment on the durability of polylactic acid/cotton -fiber biocomposites were studied by Zhang et al [11]. According to the author, the thermal and mechanical properties of a composite made from surface-treated cotton fiber and a PLA matrix are improved, and the interfacial connection between the composite's individual components is strengthened. In addition, Hussain et al. [12] evaluated the functionality of recycled polypropylene-cotton composites. Maximum values for the composite's tensile strength, modulus of elasticity, breaking strength, and flexural strength were reported by the author for fiber loadings of 0–40wt.%. As a result, many investigations into cotton fiber were conducted to better comprehend its composite properties [13, 14]. However, failure of the fiber reinforcement occurs under non-uniform loading because of the existence of voids and inadequate interfacial bonding [15]. Encapsulating the bio filler-like particles onto the composite material improves the material's interfacial bonding and gap filling, as well as its mechanical, thermal, and wear characteristics [16].

Fillers in composites are similarly crucial to the enhancement of properties. Matrixes often have fillers like biosilica, biochar, cellulose, and metallic nanoparticles implanted on them to boost their unique features. Biosilica is one of the most popular natural fillers used today; it may be derived from a broad variety of waste materials, including those generated in the food and agricultural industries as well as everyday trash. Barley husk is a source of trash in the agricultural and food processing industries. Barley, a cereal grain belonging to the grass family, is primarily cultivated in temperate climates. As of 2017, it is predicted that the global output will be 149 million tonnes [17]. The vast majority of these grains are fed to livestock. This research article examines the use of barley husk, a byproduct of the widespread cultivation of barley, to create a clean and waste-free environment. In addition, the barley husk, thanks to its high phytolith concentration, has the capacity to create silica at a rate of around 40 wt.% [18]. In addition, the annealing procedure and silane treatment are used to manufacture filler substance by decreasing the particle flaws such as inner cracking and inappropriate alignment with the molecules [19].

Due to its possible applications, biosilica is the subject of extensive scientific investigation. However, the characterization investigation of toughening natural fiber composites with functionalized barley husk biosilica was assessed by Krishnamoorthy et al [20]. Author findings reveal improved mechanical and wear qualities alongside decreased COF after including 3 vol.% of functionalized barley husk biosilica. Similarly, Babu et al. [21] investigated the epoxy based structural composite including new spinifex littoreus fiber and sugarcane biosilica. Author concluded that addition of biosilica performed as equal as synthetic form of nanosilica. In addition to this Vijayaraj et al [22] performed research on Digitariaischaemumnatural fiber along with finger millet husk biosilicaonepoxy-basedcomposite.Author reported a reduced COF of 0.266 and a wear loss of 0.013mm^3/Nm for 5 vol. % biosilica dispersion. Similar research works also done on biosilica filler and its composites [23, 24].When fillers are distributed in a high-density resin media, however, there is still room for development in terms of both dispersion ability and property enhancement. These fillers are often manufactured utilizing a thermo-chemical process, which results in physical damage, a strained grain structure, and a rough exterior. To reduce these difficulties on the ceramic fillers a conventional heat treatment method could be useful to release tension and rearrange the outside surface for better silane attachment and adhesion compatibilities. Annealing on biosilica made from biomass waste has not been reported in the literature before. In addition, there has been no research done on the impact of incorporating both biosilica and cotton microfiber into a vinyl-based resin. Thus, the present study purpose is to evaluate the impacts of annealed-silane modified biosilica on mechanical, wear, dielectric and thermal stability behavior of waste cotton microfiber-based vinyl ester composite. In addition to these characteristics, its low cost, high strength, stiffness, biodegradability, renewability, and eco-friendliness make it a promising material for usage in a variety of industries, including automotive, aerospace, household appliances, defense, space science, and recreation.

2. Experimental work

2.1. Materials

In this present study rigid vinyl ester is used as resin matrix and it has a density of 1.05 g/cm^3 and has a viscosity of 350 cps at room temperature. The vinyl ester resin was purchased from “Orson resin and coatings” Private Ltd, Mumbai, India. The matrix is prepared by adding an promoter, accelerator and catalyst namely cobalt naphthenate, 2-dimethyaniline, methyl ethyl ketone peroxide and it was procured from POLYCHEM, Chennai, India. Further, the waste cotton microfiber used as reinforcement is purchased from the nearby textile Industry and the barley husk used to make biosilica was bought from nearby food processing center. The waste cotton microfiber has length of 300-350μm with a diameter of 3-5μm. Moreover, for silane treatment process 3-Aminopropyltrimethoxysilane was used and procured from Sigma Aldrich, USA.

2.2.Extraction of annealed–surface modified biosilica

The process for extraction of biosilica from barley husk is represented in Figure 1. The procured barley husk is cleaned under distilled water for removing any dust contaminants; it then dried under oven for 2hrs at 60°C for removing wet. It is then subjected heat treatment in order to convert the husk into ash by under slow pyrolysis using muffle furnace at temperature of 200-600°C. The converted ash is fine grained add into the 1N of 1.5L of HCl solution and titrated well for 6 hrs. Now it is filtered out using Whatman filter paper to obtain residue. It is again added into the 1 N of NaOH solutions, stirred under magnetic stirrer for 2hrs at temperature of 75°C. The xerogel silica is formed; it is placed under air oven for remove the moisture. It is now fine-grained using ball milling for 15 minutes to produce a fine grained biosilica of size 60nm [25].

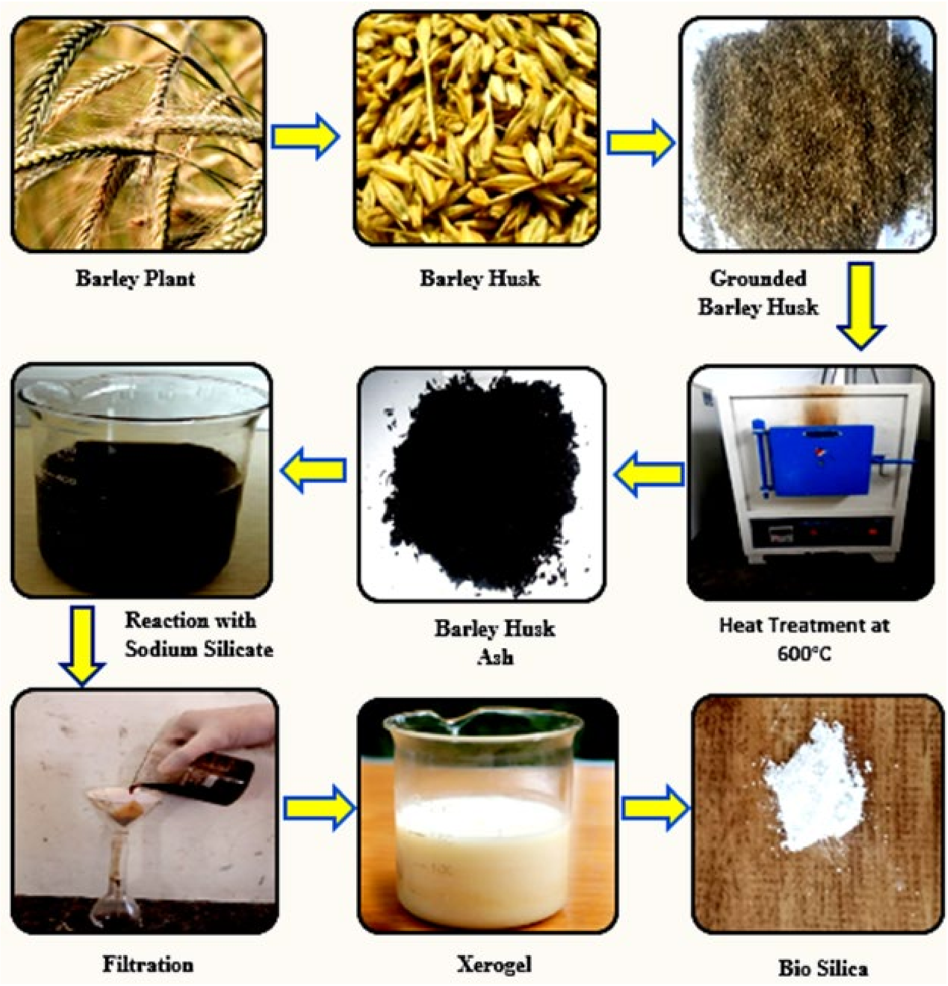

Fig. 1.Process flowchart for biosilica derived from barley husk.

The fine biosilica is further subjected to annealing process in a closed furnace up to 2h at 2000°C. The temperature was maintained for 1 h and allowed to cool in the furnace itself in a closed environment [26]. Similarly, after completing the annealing process the biosilica was treated using silane coupling agent according to the literature [27]. Figure 2 shows the X-Ray Diffraction of (a) before annealing, (b) after annealing, (c) Fourier Transform Infra Red spectra of plain biosilica and (d) Fourier Transform Infra Red spectra of surface treated(silane) biosilica. It is noted that the 2θ peaks at 21.8° in Figure 2 (a and b) indicates the silica crystals in the biosilica. The peaks resemble the amorphous nature of silica structure, which is possibly due to lower processing temperatures. However, in Figure 2 (b) a sharp peak appears in 28.1° 2θ and 40.4 °2θ indicating the transformation of three-dimensional arrays of linked tetrahedrons, each consisting of a silicon atom coordinated by four oxygen atoms as crystalline form.

Thus, the annealing process is effective in the crystalline level changes in the biosilica prepared. Finally, the FTIR spectra of as-received and silane treated biosilica shows remarkable change. In Figure 2(c) there is no functional peak in the graph provided. This indicates there is no functional groups are attached in the biosilica surface. After a successful silane treatment, the FTIR spectra shown in Figure 2(d) reveal the introduction of new functional groups. The peaks observed at 3378.09, 3285.51, and 2956.56 cm^{-1} correspond to the N-H and C-H groups introduced by the silane material. Additionally, several peaks between 1589.34 and 1072.02 cm^{-1} suggest the presence of C-H stretches likely originating from the propyl chain of the silane. The peak at

956.69 cm^{-1} indicates the Si-O-Si bond structure of the silane, which forms on the biosilica surface after the treatment. Thus, the silane treatment effectively introduces functional groups on the biosilica surface, enhancing its ability to bind with the resin medium.

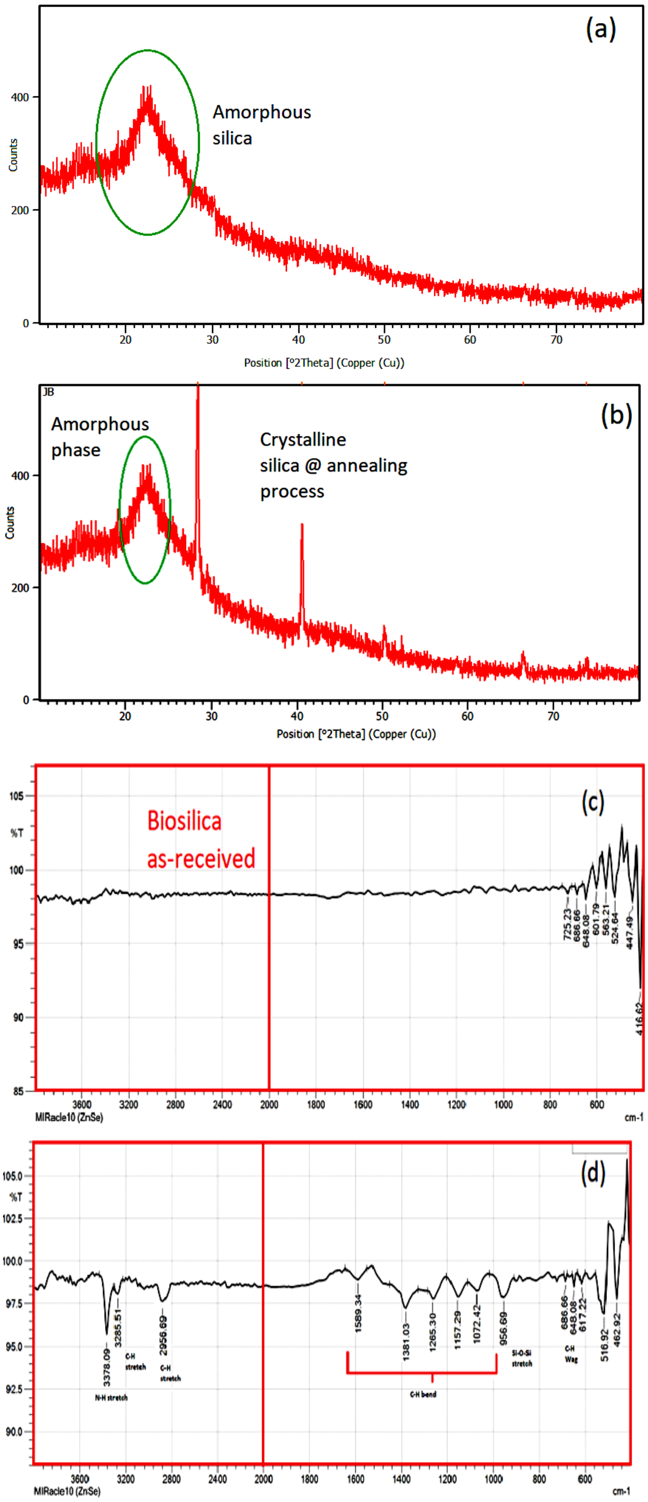

Fig. 2. XRD plot of biosilica (a) before annealing, (b) after annealing, (c) FTIR spectra of biosilica before silane treatment and (d) FTIR spectra of biosilica after silane treatment.

2.2. Preparation of composite

The present research developed a composite using annealed and silane modified biosilica, cotton fiber, and vinyl ester matrix. First vinyl ester matrix and mold are prepared. The vinyl ester matrix is developed by adding cobalt naphthenate, 2-dimethylaniline and catalyst of 2 wt.% into the resin. Now the biosilica at varying volume percent is added into the matrix and dispersed uniformly by using ultrasonicate for 15 minutes. Now the chopped cotton fiber is also added onto the matrix mixture. Finally, the fiber and filler reinforced matrix is filled into mold and allowed for curing process. The fabricated composite cured for 20 hours at room temperature and again post cured under hot air oven for 3hrs for achieving better composite performance. Table 1 composition and designation of prepared composite material [28]. Figure 3 shows the prepared composite made using the waste cotton fabric and biofiller reinforcements.

Table 1. Composite designation of vinyl ester composite.

Composite samples	Resin (vol.%)	Waste cotton fibre (vol.%)	Biosilica (vol.%)
V	100	-	-
VC	70	30	-
VCB1	69	30	1
VCB2	68	30	2
VCB3	67	30	3

Fig. 3. Post cured composite laminate.

3. Characterizations

The prepared composite performance is determined by conducting a test such Thermogravimetric Analysis (TGA), flammability, and antimicrobial. The test is assessed as per ASTM and ISO standard, and composite specimen are cut by using water jet cutting machine modelled from United States of America (USA), Maxieum, KENT type. TGA of the composite was tested by ASTM 3850 a DSC-TGA coupled analyzer (NETZSCHSTA Jupitar 409 PL Luxx, Germany) with N_2 ambience. The flammability test was done accordance with ASTM D635 with flammability standards UL-94V, vertical mode lighted by Bunsen burner. The Antimicrobial activity against the pathogenic bacteria (E. coli and S. aureus) was assessed by the zone of inhibition method (ISO 22196).The zone of bacterial inhibition around the circumference of the composite film acts as indication whether the composite possess antimicrobial properties.

4. Results and discussions

4.1. Flammability behavior

The vinyl ester matrix reinforced with annealed silane-treated biosilica and waste cotton microfibre composite material's Flammability (UL-94) tests are displayed in Table 3. With a propagation speed or velocity of propagation of 12.84 mm/min and a V-0 rating, the vertical UL-94 test demonstrated the strong flammability capacity of pure vinyl ester composite. This high flammability condition in vinyl ester is due to natural thermal degradation properties of polymer chain and its change in chemical and physical properties when subjected elevated temperature [30]. However, the cotton microfiber reinforced polymer composite shows further increased flammability and also having maximum propagation speed of 15.66 mm/min. When compared to the plain vinyl ester composite V the cotton fiber reinforced composite VC reports, higher propagation speed of 21.96% with vertical rating of V-1 grade. The reason behind such reduced flame-retardant capacity is because of high cellulose content, which is having the ability to flammable with high propagation speed [31].

Besides these, none of the composite samples shows leakages that is falling drops of the composite while flaming. Moreover, the addition biosilica shows improved flame-retardant property, and which cause reduced propagation speed of 13.19, 11.05 and 9.77 mm/min of composite VCB1, VCB2, and VCB3 respectively. Among those 3sample specimen of V, VC, and VCB3 (represented in Figure) the composite designation of VCB3 shows maximum flame retardancy and lowest propagation speed of 9.77 mm/min. When compared to fiber reinforced composite (VC), the 5 vol.% of annealed silane treated biosilica reinforced (VCB3) composite shows maximum flame retardancy and reduced propagation speed of 60.28%. this increase in propagation speed is because of the heat resisting behavior of biosilica particle [32]. Further, the silane treatment of biosilica has enhanced interfacial adhesion in the polymer composite, which makes it material more strength and high fire resisting capacity.

Table 2. Flammability behavior of various combination of vinyl ester composite.

Composite Samples	Velocity of Propagation (mm/min)	Cotton Lighten	Falling drops	Vertical standard Rating
V	12.84	NO	NO	V-0
VC	15.66	NO	NO	V-1
VCB1	13.19	NO	NO	V-0
VCB2	11.05	NO	NO	V-0
VCB3	9.77	NO	NO	V-0

Fig. 4. Burned test specimens of V, VC and VCB3 composite samples.

4.2. Thermal stability

The thermo-gravimetric analysis represented in Figure 5 provides a valuable insight into the thermal stability of different composite designations. The V composite designation displayed an initial decomposition temperature of 355°C. It's important to note that vinyl ester resins, being a type of thermosetting polymer matrix in composite materials, inherently exhibit a lower initial decomposition temperature compared to some other resin systems like epoxy [33]. The lower initial decomposition temperature is due to the presence of reactive ester groups in their chemical structure, which makes them more prone to hydrolysis and thermal breakdown. The composite's initial decomposition temperature was lowered to 320°C as a result of the addition of cotton waste fibers, which produced VC. Because cellulose, the primary organic substance found in cotton waste fibers, is more likely to break down at lower temperatures than vinyl ester resin, there is a loss in thermal stability. As the composite is subjected to heat, the cellulose within the cotton fibers begins to break down at lower temperatures, contributing to the overall decrease in the composite's thermal stability [34]. However, when annealed biosilica particles were introduced into the composites, such as VCB1, VCB2, and VCB3, a significant improvement in the initial decomposition temperature was observed. Notably, the VCB3 composite designation reached a notably high initial decomposition temperature of 388°C. The addition of biosilica particles, which raise the composites' overall thermal stability, is responsible for this improvement in TGA values. Unlike cotton fibers, biosilica particles possess better heat-conduction properties, resulting in more even heat distribution within the composite and reducing the risk of premature thermal decomposition [35]. This signifies the positive impact of incorporating biosilica particles on the composites' thermal performance, showcasing their potential for applications that demand higher thermal stability.

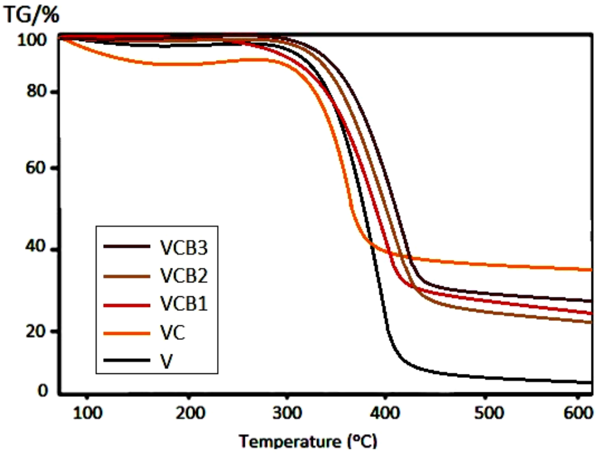

Fig.5.Thermogravimetric analysis for various composites.

4.3. Antimicrobial property

The antibacterial efficacy of several composite designations, including V, VC, VCB1, VCB2, and VCB3, against microbes, particularly S. Aureus and E. Coli, is depicted in Figure 6. The findings unequivocally show that there was no inhibitory impact of the V composite. In contrast to the V composite, the VC composite, which contained less cotton waste fiber, had slightly less antibacterial efficacy against both S. Aureus and E. Coli. Inhibitory zones for S. Aureus and E. Coli in the VC instance had sizes of 10.31 mm and 10.18 mm, respectively. The presence of cellulose in the cotton waste fibers is responsible for the inhibitory effect shown in the VC composite [36]. Cellulose are natural components and they possess antimicrobial properties and likely hindered the growth of microorganisms, resulting in the increased inhibitory zones observed in the VC composite.

Furthermore, the addition of surface modified and annealed biosilica particles increased the inhibitory zones' size even more. Increases in biosilica content (1%, 3%, and 5% vol.) resulted in greater inhibitory diameters. Consequently, the VCB3 composite showed the largest inhibitory zones, with S. Aureus measuring 14.36 mm and E. Coli at 14.72 mm. The inclusion of silane-treated biosilica particles with a greater antibacterial effect through homogeneous dispersion throughout the matrix is responsible for the antimicrobial qualities seen in the VCB1, VCB2, and VCB3 composites. The silane treatment introduces quaternary amine compounds to the surface of biosilica particles. Amines are organic compounds with a positive charge that exhibit strong antimicrobial effects [37]. Additionally, the silane-treated biosilica releases silicon ions, which also contribute to the antimicrobial properties. These ions interact with microbial cells and disrupt their physiological processes, resulting in increased inhibitory zone sizes in the composites.

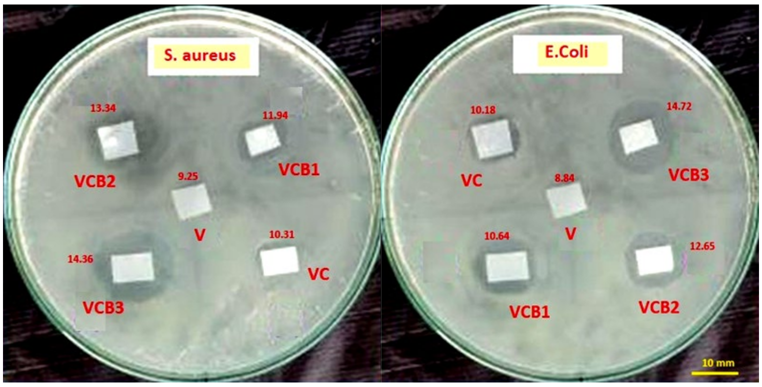

Fig. 6. Antimicrobial study on various composites.

5. Conclusions

Thus, the study concludes that reinforcement of waste biomass derived and surface modified biosilica and cotton fiber reinforcement into the polymer matrix shows better strength features such fire resistance, stabled under temperature and microbes. Based on the observation the flammability analysis results are promising. The inclusion of annealed-silane modified biosilica demonstrates considerable potential in enhancing fire resistance. Notably, the VCB3 composite achieves a V-0 rating with decreased flammability, enhanced fire retardancy, and a decreased velocity of propagation or propagation speed of 9.77 mm/min. Moreover, it shows no cotton ignition or falling drops, indicating its suitability for applications prioritizing fire safety. Furthermore, the study of thermal stability reveals that the modified composites exhibit remarkable resistance to high temperatures.

Particularly, the VCB3 composite showcases a remarkably high initial decomposition temperature of 388°C, underscoring its ability to withstand challenging thermal conditions effectively. In the antimicrobial properties, the study demonstrates the inherent resistance of these composites to microbial growth. VCB3, in line with the enhancements observed in flammability and thermal stability, exhibits improved antimicrobial characteristics. It displays increased inhibitory diameters of 14.36mm and 14.72mm against S. Aureus and E. Coli, respectively. This feature holds substantial promise for a broad spectrum of industries, particularly those emphasizing hygiene and microbial control. In conclusion, waste cotton microfiber-vinyl ester composites that contain annealed-silane modified biosilica have improved antibacterial, thermal stability, and fire resistance.

These results open exciting possibilities for the application of these advanced composites across diverse industries, including construction, transportation, and healthcare, where attributes like reduced flammability, high-temperature resistance, and antimicrobial qualities are highly valued.

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