



Experimental study on thermal and morphological analyses of green composite sandwich made of flax and agglomerated cork

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Abstract

The building sector has been showing great interest in incorporating technologically advanced materials with lightweight, ecofriendly, high strength, and stiffness properties in flooring, roofing, and partition walls, etc. In addition to the mechanical properties, these materials should have good thermal properties as well. In response to these requirements, an attempt has been made to study the thermal behavior of green composite sandwich made of flax and agglomerated cork. Composite sandwiches were fabricated by using flax as skin reinforcement and agglomerated cork as core with different densities as 240, 280, and 340 kg m⁻³ using vacuum bagging method. Glass was also used as skin reinforcement for manufacturing composite sandwiches for comparison purpose. Experiments were conducted to predict thermal properties, viz. thermal conductivity, thermal expansion, flammability, and thermal stability. The experimental results show that the lowest thermal conductivity of 0.03 W m⁻¹ K⁻¹ was observed in flax-based composite sandwich having core density of 240 kg m⁻³; the lowest thermal expansion of 29.2 × 10⁻⁵ °C⁻¹ was observed in glass-based composite sandwich having core density of 340 kg m⁻³; the highest value of time to ignition was 12 s, and minimum propagation rate was 0.25 mm s⁻¹ in flax-based composite sandwich having core density of 340 kg m⁻³; The highest initial degradation temperature was 362 °C for glass-based skin and 263 °C for cork having density of 240 kg m⁻³.

Keywords Green composite sandwich · Flax · Agglomerated cork · Thermal properties

Introduction

Energy conservation through technological development is one of the key research areas in all engineering sectors. Building sector is one among them, which consumes large

amount of energy in all regions. In buildings, a substantial share of energy goes to maintain the temperature for providing a thermal comfort. This heating and cooling load can be reduced through many ways, important being the proper design and selection of building materials [1].

The rising demand for lightweight structures in building sector has induced a significant growth in composite sandwich technology. Composite sandwich is structured by sandwiching a thick, lightweight core between two thin and stiff skins [2]. On the other hand, incorporating the natural materials in building sector has received increasing attention during recent years since the environmental concern is the major focus throughout the world [3, 4]. Composite sandwich structures are mostly made up of synthetic fibers like glass and carbon fiber as skin reinforcement and polyurethane foam and aluminum honeycomb as core. Introducing the naturally available materials as skin reinforcement and core for fabricating composite sandwich can provide benefits to the environment because of their bio-

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degradability, cost effectiveness, and natural availability. It also possesses good specific mechanical, thermal insulation, and sound absorption properties.

During last few years, many researchers have taken countless efforts to incorporate the natural fibers as reinforcement in polymer-based composites [5–10]. Flax is one among them having comparable mechanical properties with E-glass fiber [11]. Flax fibers are produced from the stems of flax plant having crystalline structure making it stronger and stiffer [12]. On the other hand, cork is a naturally available material obtained from the bark of the oak tree having remarkable properties like good shear strength, high damage tolerance to impact loads, good thermal insulation, better acoustic insulation, and excellent damping properties [13]. At present, the use of cork in flooring, wall coverings, and insulation has expanded worldwide due to the development of new cork-derived materials; the improvement of cork's characteristics; the growing importance of natural and sustainable materials as well cork's exotic character in some distant markets [14].

Few researchers have also investigated on the mechanical properties of composite sandwich made of flax as skin reinforcement and agglomerated cork as core. Sarasini et al. [15] studied the impact behavior of sandwich structures made of flax/epoxy face sheets and agglomerated cork, and they concluded that this combination could be renewable alternative to the traditional glass fiber and synthetic foam because of its peculiar mechanical behavior. Mancuso et al. [16] characterized the mechanical behavior of a green sandwich made of flax-reinforced polymer facings and cork core, and the result reported the significant mechanical properties. Apart from flax and cork, other natural materials were also incorporated in composite sandwich and their thermal behaviors were studied. Kawaski et al. experimentally investigated the thermal conductivity of the plywood-based sandwich panels with low-density fiberboard (PSW) as wood-based structural insulation materials for walls and floors. The thermal conductivity of PSW panels with densities of 340 kg m^{-3} and 410 kg m^{-3} was 0.070 and $0.077 \text{ W m}^{-1} \text{ K}^{-1}$, respectively. The authors concluded that the PSW panel presents better insulating property than the commercial wood-based boards [17]. Alavez-Ramirez et al. [18] developed a coconut fiber-filled ferrocement sandwich panel for roofing applications in schools and houses with lightweight and low thermal conductivity. Vitale et al. [19] measured the thermal conductivity of vegetable fiber-reinforced composite sandwich and compared with the synthetic counterparts. Heat transfer capacity of the composite material plays a vital role in assessment of its responsive nature to thermal energy, and building structure should be more resistive to the heat flow. Not only the thermal conductivity property, thermal

expansion, flammability, and thermal stability are also important for the building materials. Understanding the relative expansion/contraction characteristics of materials is important for application success. The difference in thermal expansion develops internal stresses and stress concentrations in the polymer, which allows premature failure to occur. Fire responsive characteristics of the composite materials are playing a significant role in the selection of the composite materials for building applications. The fire resistance property is quantified by measuring the time taken to get ignited and propagation of flame of material when ignited [20–22]. Thermal stability is also desirable for building sectors which tells mass loss over the temperature. Hence, in this paper, an attempt has been made to investigate the thermal behavior of green composite sandwich made of flax and agglomerated cork as natural materials compared with synthetic counterparts in order to explore the potential use of natural materials in building sector.

Experimental

Materials

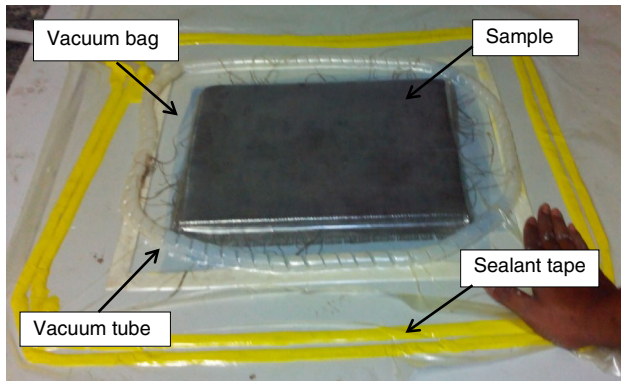
The materials used to prepare composite sandwiches were flax fiber (skin reinforcement) and agglomerated cork (core) with three different densities (240 kg m^{-3} , 280 kg m^{-3} , and 340 kg m^{-3}). Flax fiber in the form of bidirectional woven was obtained from Lineo, Belgium, having the density of 280 gsm. Agglomerated cork was obtained from Anchor Cork Pvt. Ltd., India. E-glass fiber was used as skin reinforcement for making composite sandwich for comparison purpose. Table 1 shows the physical and mechanical properties of flax fiber compared with E-glass fiber [23]. E-glass fibers in the form of bidirectional woven were purchased from Covai Seenu & Company, India, having the density of 280 gsm. The epoxy resin used for lamination was LY556 Grade with a HY951 hardener supplied by Covai Seenu & Company, India.

Sample preparation

Composite sandwiches were manufactured by vacuum bag molding process. Compared to hand layup method, the vacuum bag molding provides higher reinforcement concentrations, better adhesion between layers, and more control over resin/fiber ratios. Figure 1 shows the vacuum bag molding process. Fiber layers were placed over the mold one by one, and epoxy resin mixed with hardener was applied on each layer up to the desired bottom skin thickness. Fiber volume fraction for skin was maintained for all samples as 0.5. Agglomerated cork core was placed

Table 1 Physical and mechanical properties of flax and E-glass fiber

Fiber type	Diameter/ μm	Density/ g cm ⁻³	Tensile strength/ MPa	Elastic modulus/ GPa	Specific modulus/ GPa g ⁻¹ cm ³	Elongation at failure/ %
Flax	12–600	1.4–1.5	343–2000	27.6–103	45	1.2–3.3
E-glass	< 17	2.5–2.6	2000–3500	70–76	29	1.8

**Fig. 1** Vacuum bag molding process

on the completed bottom skin. The top skin was stacked up like the bottom skin on top of the core. After the layup was fabricated on mold from precut plies of fabric, core and resin, a non-adhering film of vacuum bag was placed over the layup and sealed at the mold flange. A vacuum pump was used to evacuate the air inside the vacuum bag and compress the part under atmospheric pressure for the compacting and hardening process to take place. After 24 h, the cured composite sandwiches were removed from the vacuum bag. Table 2 presents the different combinations of composite sandwiches manufactured and tested.

Thermal conductivity

Thermal conductivity is essential to quantitatively compare the different thermal insulation materials. Heat flow meter (HFM)—HFM 436/3 Lambda, was used to measure the thermal conductivity of the composite sandwich according to ASTM Standard C518. Figure 2 shows the heat flow meter for measuring the thermal conductivity. A 300 mm × 300 mm sample with a thickness of 13 mm was placed between hot and cold plates, and the heat flow was created by the temperature difference. Precise load on the specimen ensures that the plates make intimate contact with the specimen across the entire surface in order to produce a uniform contact resistance. The guarded area ensures 1-D heat transfer. With the heat flux (q), the temperature difference across the specimen (ΔT) and the thickness of the specimen (L), the thermal conductivity

(k) of the specimen can be calculated using Fourier equation (1).

$$k = \frac{qL}{\Delta T} \quad (1)$$

Thermal expansion

The dimensional stability of the materials is compared by coefficient of thermal expansion (CTE), and materials are expected to have least thermal expansion when subjected to thermal load. Otherwise, the induced thermal stress at the structural joints creates the buckling effect which is undesirable for structural composites. The thermal expansion of composite materials was found by using experimental rig consisting of vacuum oven and digital micrometer as shown in Fig. 3. Initially, the specimens were prepared from the sandwich panels as per the dimensions (90 mm × 13 mm × 13 mm). Initial lengths of specimens were measured using digital micrometer, and the points were marked at that place. Then, the specimens were placed inside the vacuum oven and their temperature was raised to 90 °C and it was maintained at that temperature till steady-state condition was reached. Finally, the specimens were taken out and measured for final length using digital micrometer. The CTE (α) for a material is expressed in Eq. (2),

$$\alpha = \frac{\Delta L}{L\Delta T} \quad (2)$$

where ΔL is change in length, L is the original length of the sample, and ΔT is change in temperature.

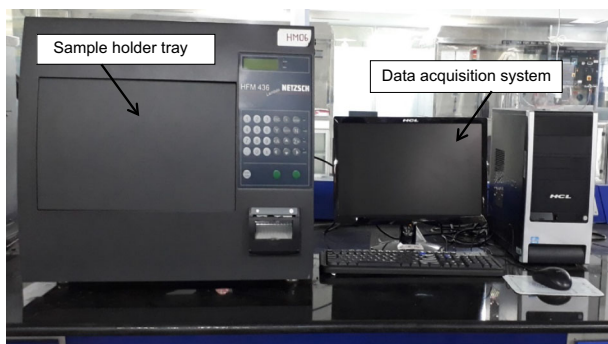
Flammability

Flammability is the ability of a material to burn in fire when it is exposed to such an environment. The samples were prepared with dimensions of 125 mm × 13 mm × 13 mm. In this test, the time to catch fire and the flame propagation rate for the samples were inferred both in vertical as well as horizontal tests. Experimental setup of vertical and horizontal flammability is shown in Fig. 4. Flame was created using methane gas. In vertical flammability test, the top of the test specimen is clamped to

Table 2 Different combinations of composite sandwiches

S. no.	Sample name*	Composite sandwiches		Layer sequence*	Skin thickness/mm	Core thickness/mm	Sandwich thickness/mm
		Skin	Core with density				
1	FEC 240	Flax/epoxy	Cork 240 kg m ⁻³	2FE/C240/2FE	1.5	10	13
2	FEC280	Flax/epoxy	Cork 280 kg m ⁻³	2FE/C280/2FE	1.5	10	13
3	FEC340	Flax/epoxy	Cork 340 kg m ⁻³	2FE/C340/2FE	1.5	10	13
4	GEC240	Glass/epoxy	Cork 240 kg m ⁻³	3GE/C240/3GE	1.5	10	13
5	GEC280	Glass/epoxy	Cork 280 kg m ⁻³	3GE/C280/3GE	1.5	10	13
6	GEC340	Glass/epoxy	Cork 340 kg m ⁻³	3GE/C340/3GE	1.5	10	13
7	GFEC 240	Glass/flax/epoxy	Cork 240 kg m ⁻³	GE/FE/C240/FE/GE	1.5	10	13
8	GFEC 280	Glass/flax/epoxy	Cork 280 kg m ⁻³	GE/FE/C280/FE/GE	1.5	10	13
9	GFEC 340	Glass/flax/epoxy	Cork 340 kg m ⁻³	GE/FE/C340/FE/GE	1.5	10	13

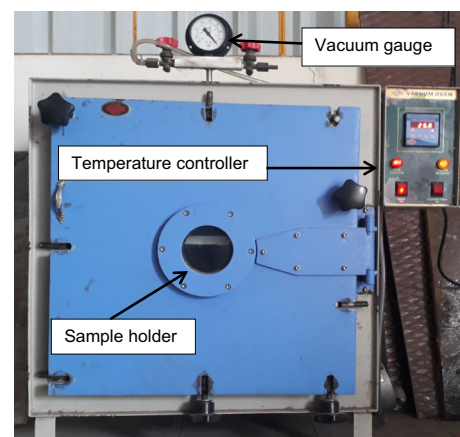
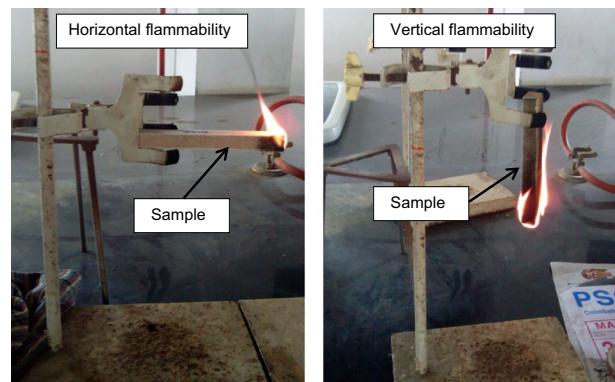
*F—flax, G—glass, E—epoxy, C—agglomerated cork, 240, 280, and 340—density of the cork in kg m⁻³

**Fig. 2** Heat flow meter test rig

a stand and the burner is placed directly below the tip of the specimen. In horizontal flammability test, the specimens are clamped in horizontal direction and the flame is applied at the free end. In both cases, flame is exposed to the tip of the sample. The flame is brought into contact with the test specimen for 10 s, after which the burner is removed. The flame should be a blue flame and 20 mm in height.

Thermal stability

The stability of the structural composite sandwich against the thermal load is very much important as the dimensional stability of the material against the mechanical load is directly influenced by the material loss due to thermal decomposition. Thermal stability of the materials can be found out by thermogravimetric analysis (TGA) by means of finding out the degradation of material as a function of temperature in a controlled environment. It was executed by a thermogravimetric analyzer of NETZSCH STA 449 F3 Jupiter as shown in Fig. 5. In order to analyze the thermal stability of the composite sandwich samples, the skin and core involved in the samples were tested individually. The samples to be tested were extracted from the

**Fig. 3** Experimental test rig for thermal expansion measurement**Fig. 4** Experimental setup for flammability

skin as well as from the core materials. Samples weighing 5 mg were placed in a ceramic crucible, and tests were carried out in nitrogen atmosphere at the heating rate of 20 °C min⁻¹ between 25 and 600 °C.

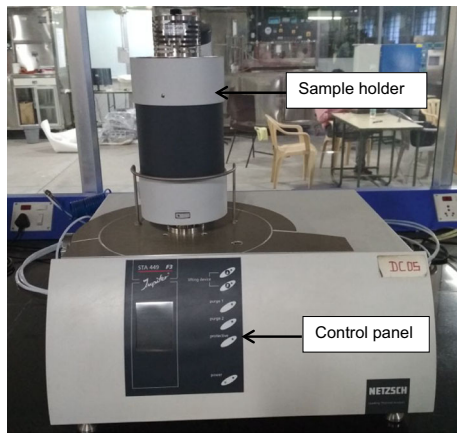


Fig. 5 Thermogravimetric analyzer

Morphological test

The morphology and structure of composite sandwiches were examined by scanning electron microscope (SEM) (ZEISS Sigma) at an accelerating voltage of 10 kV. The samples were sputter-coated with gold, and images were captured at various magnifications ranging from 150X to 500X. Microscopic images of cork having varying densities were captured using Stereo Microscope (ZEISS Stemi 508) at a magnification range of 6.3X.

Results and discussion

Experiments were conducted to quantify the thermo-physical response of the composite sandwich, and the observations were compared based on different properties found experimentally.

Analysis of thermal conductivity

The composite sandwich, which responds poorly to heat transfer (conduction), befits a dimensionally stable material suitable for the structural applications. The thermal conductivity of the different composite sandwiches is shown in Fig. 6. From the experimental results, it is inferred that flax fiber-reinforced composite sandwich has lower thermal conductivity among the composite sandwich samples taken for the study.

It is observed that flax fiber-reinforced composite sandwich (FEC) sample has 39–48% and 35–47% lower thermal conductivity than glass fiber-reinforced composite sandwich (GEC) and glass-flax fiber-reinforced composite sandwich (GFEC), respectively, irrespective of density of core. The FEC is comparatively less conductive than GEC. So, the thermal resistance offered by the FEC is more and GFEC shares both advantages and centered. The lower

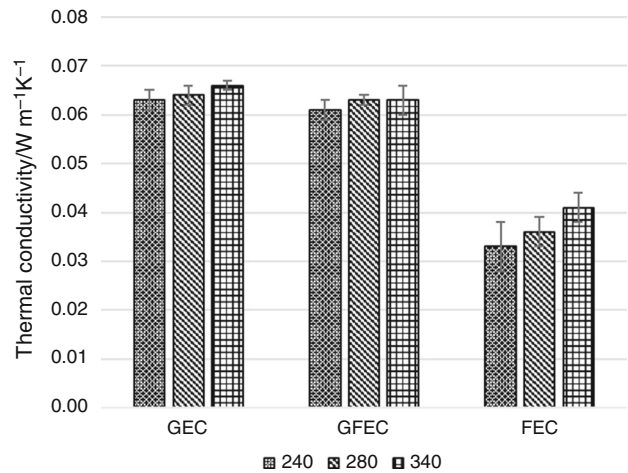


Fig. 6 Thermal conductivity of composite sandwich

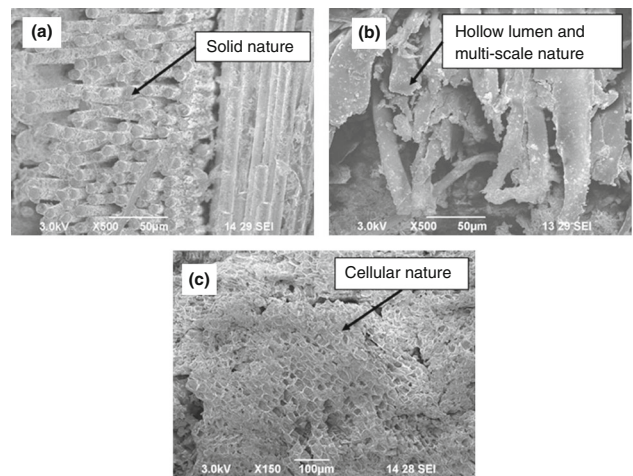


Fig. 7 SEM images of a glass fiber, b flax fiber, c agglomerated cork

thermal conductivity of FEC is due to the porous nature of flax fiber reinforcement, and the light density of the flax fiber offers large space for the entrapment of air between the fibers [24]. Figure 7 shows the SEM images of glass fiber, flax fiber, and agglomerated cork; glass fiber that has solid nature marks an increase in thermal conductivity, flax fiber that has hollow lumen nature helps to make good thermal resistance, and agglomerated cork that has cellular nature helps to reduce the heat transfer, respectively. Also, the contact resistance due to different electron affinity between the skin and core material is higher which ensured the lower thermal conductivity of FEC sample. Apart from the skin reinforcement material, the core material density also greatly influences the thermal conductivity of composite sandwiches.

FEC 240 has 8% and 19% lower thermal conductivity than FEC 280 and FEC 340, respectively. Figure 8 shows microscopic images of the cork samples which helps to realize the packing density of samples. In cork having the

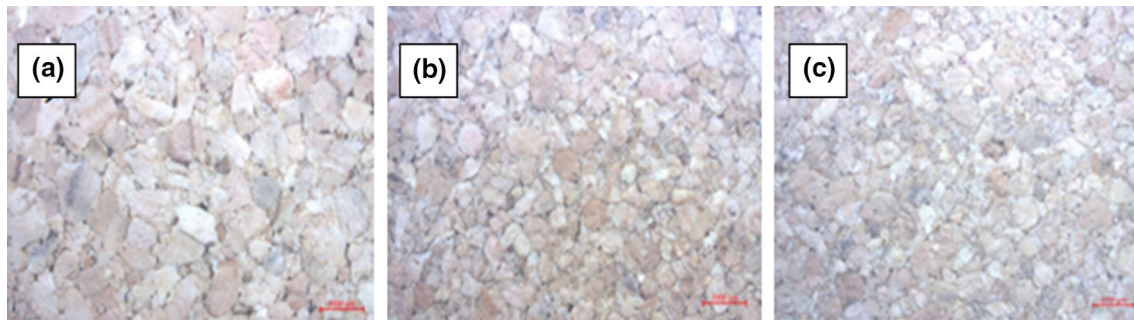


Fig. 8 Microscopic images of agglomerated cork. **a** Cork 240 kg m^{-3} , **b** cork 280 kg m^{-3} , **c** cork 340 kg m^{-3}

density of 240 kg m^{-3} , particles are loosely packed and it is having more space between particles. The lower the density of cork, the higher the cavity space, which in turn offers a large conductive resistance to the heat flow [13]. But, the higher density of cork offers feasibility to transfer the thermal energy through lattice vibration. Among all sandwich samples, the flax fiber-reinforced sandwich sample having low density of cork core (FEC 240) acts as better thermal insulator for building applications.

Analysis of thermal expansion

Figure 9 shows the coefficient of thermal expansion of composite sandwiches. The experimental results expressed that the GEC samples possess good dimensional stability than GFEC and FEC samples, and it exhibited lower thermal expansion when exposed to thermal load. It is quantified that the coefficient of thermal expansion of GEC samples is 2–8% and 4–11% lower than GFEC and FEC samples, respectively, irrespective of core density. Flax fibers are porous in nature which allows the expansion when subjected to thermal load. But, in the glass fiber, having higher thermal conductivity helps in dissipation of heat which results in reduced thermal expansion. The effect

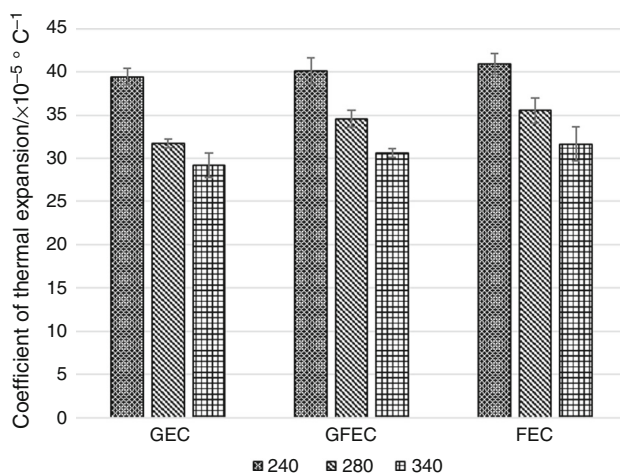


Fig. 9 Coefficient of thermal expansion of composite sandwich

of skin in thermal expansion of composite sandwich is quite minimum, but it is mostly influenced by the core material density. Composite sandwich samples having cork core density of 340 kg m^{-3} have 8–11% and 23–26% lower coefficients than the samples having cork core densities of 280 kg m^{-3} and 240 kg m^{-3} , respectively, irrespective of skin reinforcement. This is because of high-density core material facilitating reduced thermal expansion by the presence of more granules. This result depicts that GEC having cork core density 340 kg m^{-3} has lower coefficient of thermal expansion, and it provides good dimensional stability than other samples.

Analysis of flammability

The performance of composite sandwich when exposed to fire was tested in both vertical and horizontal directions, and the results were compared. Both vertical and horizontal test results exhibited quite similar response when composite sandwich was exposed to the fire. The ignition time is slightly lower for horizontal test than the vertical test because of the less exposing area to the flame, and the same was ensured from the experimental results. From Fig. 10, it is observed that the GEC samples having cork core density of 240 kg m^{-3} take lesser time to ignition and it is ensured that it catches fire within 4 s when exposed to flame. The GFEC and FEC samples have 7 s and 8 s, respectively, for the same 240 kg m^{-3} core density. In all sandwich samples, the low ignition time is experienced due to the presence of epoxy which catches fire easily before the fibers and core materials because of its poor self-ignition temperature [25]. But the presence of fiber in the composite resists the fire propagation in all composite sandwich samples. In GEC samples, when the epoxy starts igniting, the glass fiber is immediately separated which in turn offers poor resistance to the flame. But in case of FEC samples, the flax observed the moisture in the epoxy and offers good resistance compared to GEC samples.

The presence of agglomerated cork core in the composite sandwich possess low burning time, but the fire

Fig. 10 Time to ignition of composite sandwiches

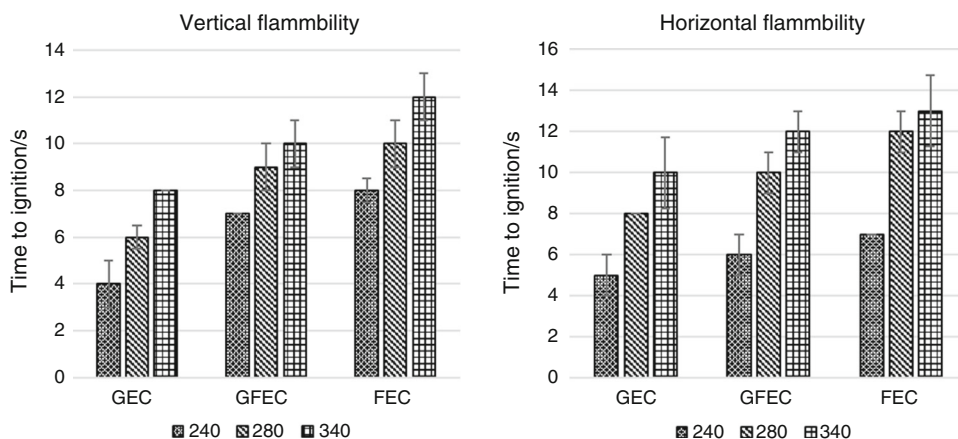
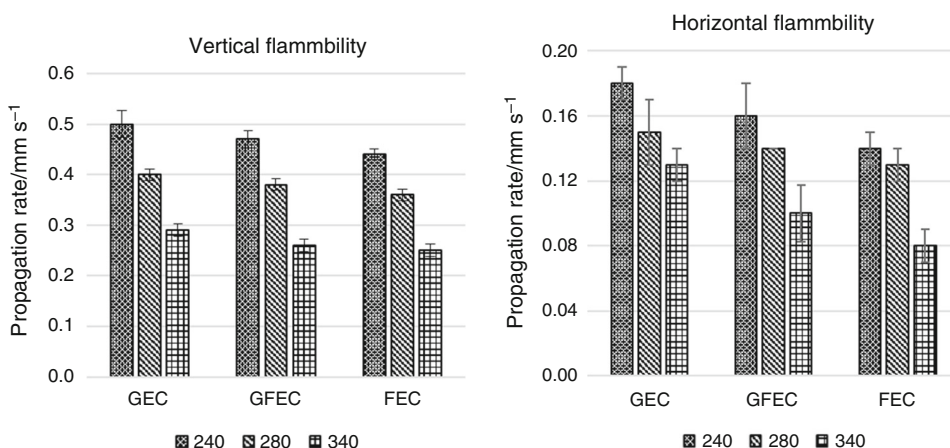


Fig. 11 Propagation rates of composite sandwiches



starts from external skin and penetrate it more easily. The high-density agglomerated cork offers less space for the internal burning of core material because of the closely packed grains [13]. The same is experienced by GEC, GFEC, and FEC samples. So, it can be concluded that FEC with 340 kg m⁻³ of core density has the highest resistance to flame among all the samples.

The flame propagation rate ensures the fire resistance property of the composite sandwich. The same is experimented and plotted for vertical and horizontal directions as shown in Fig. 11. The horizontal test and vertical test show a similar trend in propagation rate, but the horizontal test shows lower value because of less exposing area. The GEC samples experienced high propagation rate compared to GFEC and FEC samples irrespective of core density. This is because of glass fiber segregation from the epoxy resin during experimentation that permits the fire to propagate quickly through the epoxy resin itself. FEC sample having cork core density of 340 kg m⁻³ has the least propagation rate of 0.08 mm s⁻¹ which is 38% and 20% lesser than GEC and GFEC having cork core density of 340 kg m⁻³, respectively. In FEC samples, the epoxy forms a complex

interface with flax fiber and thus reduces the propagation rate. Like the ignition time property, the propagation rate also reduces when the density of the core increases in all samples irrespective of skin reinforcement. From the experimental results, it is observed that increasing the core density of FEC samples from 240 to 340 kg m⁻³ reduced flame propagation rate about 42%. This is because of high accumulation of cork granules in core leading to reduction in the flame propagation.

Analysis of thermal stability

The TGA for different skin materials was conducted, and the results are depicted in Fig. 12. In flax–epoxy (FE) skin, the degradation of material started at the temperature of 333 °C and the loss of mass is found only 9% up to the initial degradation temperature. This is identified due to the evaporation of moisture and other volatile contents present in the flax fiber. From 333 to 469 °C, the major amount of mass is lost around 71% because of the thermal degradation of hemicellulose, wax, and pectin present in the flax. The thermal stability of the epoxy is higher, and the

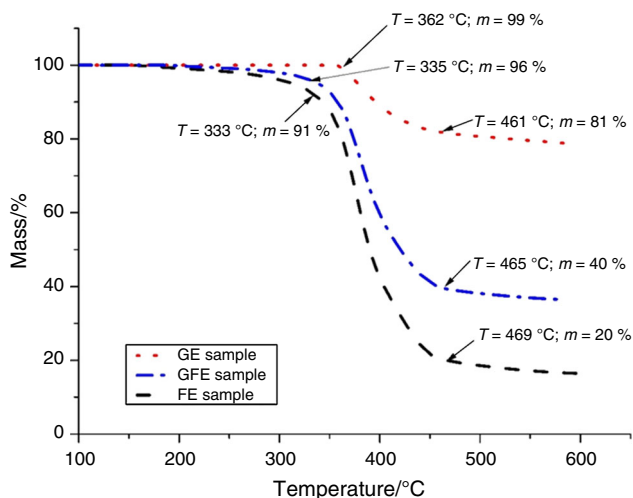


Fig. 12 TGA curve for different skin materials

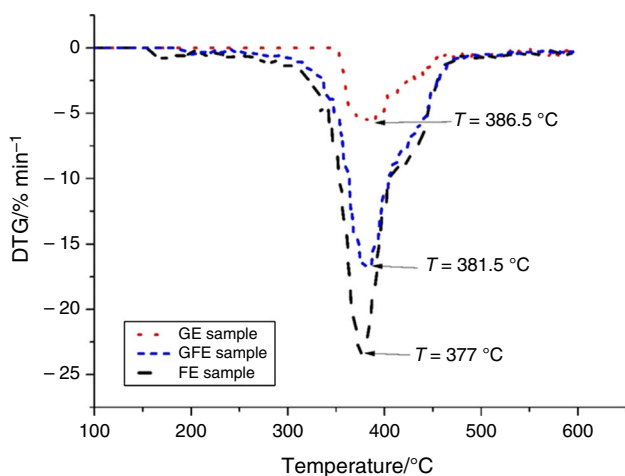


Fig. 13 DTG curve for different skin materials

presence of flax in the skin reduces the overall thermal stability by losing its ingredients.

Figure 13 shows the differential thermogravimetry (DTG) curve of different skin materials. With the help of DTG curve, the major degradation for FE skin was found at the temperature of 377 °C. After the final degradation, residues were present in the form of lignin around 20%. The glass–epoxy (GE) skin expressed better thermal stability by ensuring higher initial degradation temperature of 362 °C, and mass loss experienced is less than 1%. The major degradation of GE was observed in the temperature range of 362–461 °C, and residues presence was about 81%. From the DTG curve, the maximum percentage of loss is experienced at the temperature of 386.5 °C. The glass/flax–epoxy (GFE) skin degradation starts at 335 °C with mass loss of 4% and the degradation happened in the temperature range of 335–465 °C and the DTG curve shows that the maximum mass reduction was experienced

Table 3 Degradation temperature of skin and core materials

Samples	Initial degradation temperature/°C	Final degradation temperature/°C	Char residue/%
Flax/epoxy (FE)	333	469	20
Glass/flax/epoxy (GFE)	335	465	40
Glass/epoxy (GE)	362	461	81
Cork 240 kg m ⁻³ (C240)	263	487	37
Cork 280 kg m ⁻³ (C280)	245	494	29
Cork 340 kg m ⁻³ (C340)	231.5	516	28

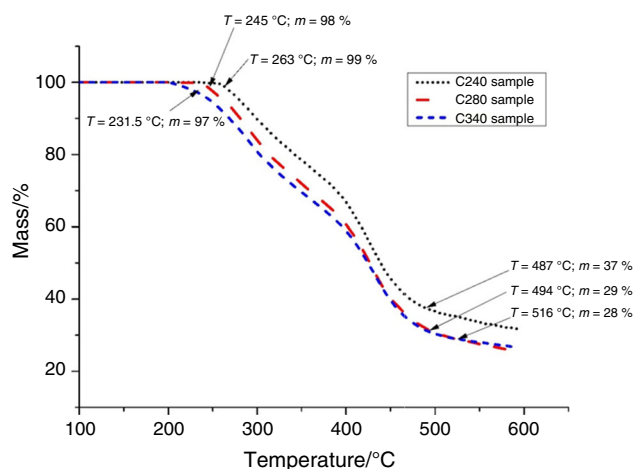


Fig. 14 TGA curve of agglomerated cork core with different densities

at the temperature of 381.5 °C. Table 3 shows the degradation temperature of different skin and core materials.

Figure 14 represents the TGA curve of agglomerated cork core material having density of 240, 280, and 340 kg m⁻³. The TGA curve of all different cork densities has quite similar trends. The loss of mass was minimal for the temperature below 200 °C, and this is because of the moisture desorption which is directly influenced by the core density. The degradation of core material was initiated at 263 °C, 245 °C, and 231.5 °C for different core densities of 240, 280, and 340 kg m⁻³, respectively. A major degradation is observed invariably in the range of 230–520 °C for all densities of the core material selected, and the loss of mass is observed around 70%. The final degradation of the core material is found to be 487 °C, 494 °C, and 516 °C for core density of 240, 280, and 340 kg m⁻³, respectively. The presence of hemicellulose in the core material degraded, which cause major loss of mass, and the char residue left was about 28–37% of initial mass. Figure 15 shows DTG curve clearly indicating the

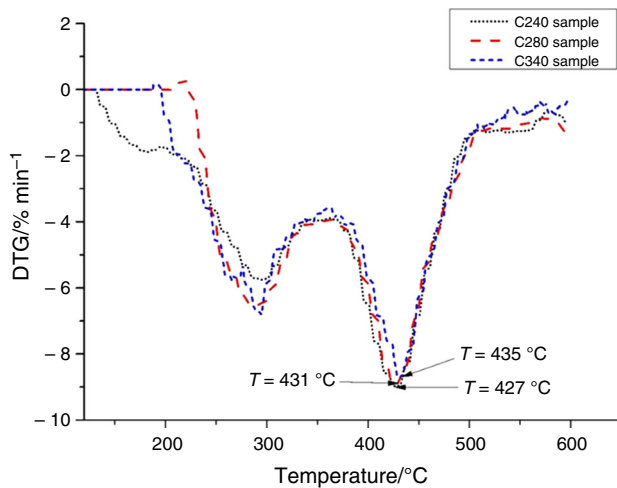


Fig. 15 DTG curve of agglomerated cork core with different densities

two-step degradation for all three core densities and the maximum reduction in mass occurs in the temperature range of 427–435 °C.

Conclusions

The experiments were conducted to reveal the thermal conductivity, thermal expansion, flammability, and thermal stability of the samples based on which the performance of the composite sandwich was studied. The performance output expected is lower thermal conductivity, lower thermal expansion, longer time to ignite, lower propagation rate, and higher thermal degradation temperature.

The results reveal that FEC having cork core density of 240 kg m⁻³ has the lowest thermal conductivity of 0.03 W m⁻¹ K⁻¹, whereas FEC having cork core density of 340 kg m⁻³ has 19% higher thermal conductivity and GEC has nearly 50% higher thermal conductivity. The lowest thermal expansion of 29.2×10^{-5} °C⁻¹ has been obtained for GEC having cork core density of 340 kg m⁻³. The thermal expansion of FEC does not vary much and is in the range of 4–11% higher than that of GEC. In the flammability test, FEC having cork core density of 340 kg m⁻³ takes more time to ignite (12 s) when compared to GEC (4–8 s). The propagation rate is minimum for FEC having cork core density of 340 kg m⁻³ at the rate of 0.25 mm s⁻¹, while the propagation rate for GEC is in the range of 0.44–0.5 mm s⁻¹. Hence, FEC having cork core density of 340 kg m⁻³ has got better flame-retardant property than its counterparts. The thermal stability is determined for the skin and core separately. The thermogravimetric results reveal that the GE skin has better thermal stability than FE skin, as glass has silica in it and the initial degradation temperature is highest at 362 °C. It

is evident from the study that the initial degradation temperature for cork 240 kg m⁻³ is higher (263 °C) when compared to cork 280 kg m⁻³ and 340 kg m⁻³. The results got from experimental tests reveal that composite sandwich performance essentially depends on the type of fiber, core, resin materials, core density, and cork granule size. These variables can be adjusted in function of the final application intended for the composite sandwich.

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Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest with respect to the research, authorship, and/or publication of this article.

Ethical approval This article does not contain any studies with human participants or animals performed by the author.

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