Composites Science and Technology 117 (2015) 62–68

Contents lists available at ScienceDirect

Composites Science and Technology

journal homepage: http://www.elsevier.com/locate/compscitech

Characterization of high glass transition temperature sugar-based epoxy resin composites with jute and carbon fibre reinforcement

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ARTICLE INFO

Article history:
Received 18 March 2015
Received in revised form
31 May 2015
Accepted 3 June 2015
Available online 6 June 2015

Keywords:
Glucose-based epoxy
Carbon fibres
Mechanical properties
Sandwich structures
Dynamic mechanical thermal analysis

ABSTRACT

The applicability of a newly synthesized glucose-based renewable epoxy resin component was tested in jute fibre and carbon fibre reinforced composites, respectively. Dynamic mechanical properties, glass transition temperature, tensile and bending strength and modulus was determined and compared to the properties of mineral oil based benchmark materials: aromatic bisphenol-A based, aliphatic glycerol and pentaerythritol based epoxy resin composites. For potential aircraft interior applications sandwich structures were prepared using jute fibre reinforcement and polymethacrylimide foam as core. According to the results the novel glucose-based epoxy monomer can be an alternative to the commonly used diglycidyl-ether of bisphenol-A (DGEBA), including high-temperature composites applications up to 160 °C.

1. Introduction

The tendency towards substitution of mineral oil based polymers by bio-based ones has emerged also in polymer composite industry, including demanding sectors as automotive and aircraft applications [1]. Besides the environmental advantages, the partial or full replacement of polymer matrices and reinforcements by renewable ones can be a strategy to reduce the dependence on petrochemicals and eliminate the effect of their fluctuating price level as well. In recent years several attempts were made to find an appropriate substitute for the most widely used thermoset composite matrix material, diglycidyl-ether of bisphenol-A (DGEBA) epoxy monomer. Currently, one of the most common ways to synthesize bio-based epoxy resins (EPs) is the epoxidation of different vegetable oils, which are principally fatty acid esters of glycerol [2–6]. However, due to the long aliphatic chains of these vegetable oils, their low functionality and consequent low crosslink density, the cured EPs have lower glass transition temperature values (T_g) and worse mechanical properties than DGEBA [7,8]. In order to enhance these properties, the next reasonable step is to mix bio-based epoxidized oils with petrol based EPs, mostly with DGEBA [9–14]. More promising results were achieved with epoxy monomers synthesized from polysaccharides, such as cardanol [15,16], isosorbide [17], sucrose [18] or rosin [19]. In the case of the cardanol based matrix [16], carboxyl-terminated polybutadiene liquid rubber was added to increase the tensile properties. In isosorbide based EPs Chrysanthos et al. [17] reached improved storage modulus below the T_g, but the T_g values were 50 °C lower than the DGEBA based reference. In case of sucrose-based EPs prepared by Sachinvala et al. [18] the break shear and Young's modulus values were similar to the values of DGEBA. The T_g of epoxy-allyl-sucrose was below, while the T_g of epoxy-crotil-sucrose was in the same range as the T_g of DGEBA. Liu et al. [19] prepared EP components from rosin with high T_g and flexural strength close to DGEBA's.

In order to prepare fully bio-based composites, not only the polymer matrix, but also the reinforcement should be renewable. From the wide range of natural fibres suitable for this purpose jute is one of the most promising bio-based reinforcing materials, due to its high cellulose content and relatively good mechanical properties compared to other natural fibres, furthermore it is produced in large quantities worldwide [20]. Nevertheless, the literature on all-bio composites made from jute fibres and bioresins is limited, mainly dealing with epoxidized plant oil composites. Avancha et al. [21] prepared jute reinforced soy resin biocomposites. Best mechanical properties (tensile strength of 35 MPa and tensile modulus of 1546 MPa) were reached with composites consisting of 60 mass%

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http://dx.doi.org/10.1016/j.compscitech.2015.06.001
0266-3538/

jute reinforcement and 40 mass% soy resin compounded with 7 mass% furfuraldehyde. Ramamoorthy et al. [22] compared the properties of acrylated epoxidized soybean oil composites reinforced with jute mat, regenerated cellulose mat and glass fibre. The jute biocomposites had a tensile strength of about 50 MPa and tensile modulus of about 10 MPa. Tensile, flexural and impact properties could be improved by hybridization with glass fibre and cellulose. Manthey et al. [23] prepared jute biocomposites from blends of epoxidized hemp oil (EHO) and epoxidized soybean oil (ESO), respectively, with DGEBA. EHO and ESO jute-based samples displayed similar tensile behaviour at a concentration of 10 mass% bioresin, a significant reduction in mechanical properties occurred after 30 mass% bioresin content. Niedermann et al. [24] investigated jute reinforced biocomposites from ESO and their blends with DGEBA, a glycerol- and a pentaerythritol-based aliphatic epoxy resin. Up to 25 mass% ESO-content the mechanical properties were comparable to the reference composites'. Campaner et al. [25] manufactured composite pipes from an EP crosslinked with a cardanol based novolac as matrix and jute fibres by filament winding technology, and carried out tensile and parallel plate compression tests on the composite pipes.

According to the literature review, although there are some promising results in field of bio-based EP composites, the breakthrough leading to massive application in more demanding sectors as aircraft industry is yet to come.

In this study a newly synthesized high glass temperature glucose based (**g**luco**f**uranoside based **t**rifunctional **e**poxy– GFTE) EP component [26,27] was tested as polymer matrix with amine and anhydride type curing agent. As reference resins, petrol oil based aromatic DGEBA, aliphatic glycerol based (GER – **g**lycerol based **e**poxy **r**esin) and pentaerythritol based (PER – **p**entaerythritol based **e**poxy **r**esin) EP monomers were used as matrices. The latter two are currently synthesized on mineral oil base, but can be potentially synthesized from renewable sources (glycerol is available in large quantities from natural fatty acids, while pentaerythritol can be produced from bio-based methanol as well). As natural sourced reinforcement woven jute fabric was chosen. According to the previous results [24,28], before the composite manufacturing no alkali treatment was applied on the woven jute fabric. As reference reinforcement woven carbon fabric (CF) was used.

The aim of this work was to compare the thermomechanical and mechanical properties of jute and carbon fibre reinforced biocomposites prepared from newly synthesized, renewable sugar-based epoxy resin to mineral oil based benchmark (DGEBA, PER, GER) composites and to test its applicability in sandwich composite structures for potential aircraft interior flooring applications.

2. Materials and methods

2.1. Materials

As polymer matrix a novel, renewable, glucofuranoside based trifunctional EP monomer (GFTE) with 160 g/eq epoxy equivalent weight (synthesized and characterized previously by the research group of the authors [26,27]) was used. As reference resin conventional mineral oil based aromatic bifunctional DGEBA with 188 g/eq, aliphatic trifunctional glycerol (GER) with 144 g/eq and tetrafunctional pentaerythritol based (PER) with 168 g/eq epoxy equivalent weight were used, provided by IPOX Chemicals Ltd. (Budapest, Hungary).

These EP components were cured by anhydride type methylhexahydrophtalic-acid (MHHPA) with 1-methylimidazole catalyst and amine type diethylene-toluene-diamine (DETDA) curing agents. The anhydride type (AR917 – trade name: Aradur 917) curing agent with 1-methylimidazole catalyst (trade name: DY070) was acquired from Huntsman Advanced Materials (Basel, Switzerland). The anhydride equivalent value (160 g/eq) was calculated from the molecular mass. The accelerator was applied in 2 mass% related to the mass of the EP component. This pthalic anhydride based component can be potentially also synthesized from natural sources [29], offering the possibility of a fully bio-based matrix material. The amine type curing agent (DETDA – trade name: DETDA80) was acquired from Lonza (Basel, Switzerland). The amine hydrogen equivalent value of the amine curing agent was 45 g/eq. During the sample preparation in all cases stoichiometric ratio of EP component and hardener was used. Based on previous results on these matrices [27] in case of AR917 the curing cycle consisted of two steps: 2 h at 100 °C and 2 h at 140 °C, while in case of DETDA 1 h at 100 °C, 1 h at 150 °C and 2 h at 175 °C heat treatment was applied. The bio-based/potentially bio-based/synthetic content (mass%) of the GFTE matrices was in GFTE-AR917 system 50/49/1, in GFTE-DETDA system 78/0/22 mass%.

As reinforcement raw linen jute fabric with 270 g/m^2 areal weight (provided by Műszaki Konfekció Kft. (Szeged, Hungary)) and UDO® MX CST 200 type biaxial CF fabric with 200 g/m^2 areal weight (from SGL Technologies GmbH (Wiesbaden, Germany)) were used. The bio-based/potentially bio-based/synthetic content (mass%) of the GFTE composites was in GFTE-AR917 jute system 80/20/0, in GFTE-DETDA jute system 91/0/9, in GFTE-AR917 carbon composite 20/20/60, in GFTE-DETDA carbon composite 20/0/80.

As core material for the jute reinforced sandwich composite structure Rohacell XT110 type closed-cell rigid polymethacrylimide foam (obtained from Evonik Industries (Essen, Germany)) with 110 kg/m^3 density was used in two different thicknesses (6.5 and 20 mm).

2.2. Methods

2.2.1. Composite preparation

Jute and CF fabric reinforced composites were prepared from GFTE, DGEBA, GER and PER EP components, cured both with AR917 and DETDA. For the preparation of composites 6 layers of jute fabric, 8 layers of CF fabric were used in [0/90$^f_8$] layer order, respectively, in order to reach the same laminate thickness. The laminate size was 140 × 140 × 2 mm (length x width x thickness) in each case. Prior to composite preparation, the jute fabric layers were dried at 80 °C for 2 h under 50 mbar vacuum in a Sheldon Manufacturing (Cornelius, USA) 1465 vacuum oven. The composite laminates were made by hand lamination in a press mould and then these laminates were put under compression with 200 bar hydraulic pressure in a Collin Teach-Line Platen Press 200E (Dr. Collin GmbH, Munich, Germany) press to achieve high and uniform fibre content in the composites. The heat treatment of the laminates (same as curing cycles detailed in 2.1. Materials section) was carried out during the pressing. The measured fibre content was approximately 59–61 mass% in every case.

In case of the sandwich composites consisting of GFTE or DGEBA matrix, cured with AR917 and reinforced with jute fabric, a spacer element was placed between the pressing platens to avoid the unnecessary compression of the core foam material. For the top and the bottom composite layer, two jute fabric layers were impregnated with EP systems.

2.2.2. Characterization of the composites

2.2.2.1. Dynamic mechanical thermal analysis (DMTA). DMTA measurements were carried out with Q800 DMTA device from TA Instruments (New Castle, DE, USA) in three point bending setup in the range of 0–200 °C, with 3 °C/min heating rate, with 50 mm span length. The specimen size was 55 × 10 × 2 mm

(length x width x thickness) in every case. The frequency was 1 Hz, the amplitude was strain controlled with 0.1% relative strain. From the results, storage modulus (E′) and tanδ curves, storage modulus values on different temperatures (T) and glass transition temperature (T_g) values were determined by the software of the DMTA device (TA Instruments Universal Analysis 2000 4.7A version).

2.2.2.2. Tensile test. Tensile tests were carried out to determine the composites tensile strength (σ_m) and tensile modulus values (E_m) by a Zwick Z020 (Ulm, Germany) type computer controlled universal tester, used with a 20 kN capacity load cell and parallel vice grips. Based on EN ISO 527 the specimen size was 140 × 10 × 2 mm (length x width x thickness). The test speed was 2 mm/min, and the initial test length was 100 mm. The temperature was 22.9 °C and the relative humidity was 52%. During the test, force and displacement values were recorded and the tensile parameters (σ_m, E_m) were calculated according to the standard.

2.2.2.3. Bending test. Bending tests were carried out in three point bending setup to determine the composites bending strength (σ_{fm}) and bending modulus values (E_{fm}) by a Zwick Z020 (Ulm, Germany) type computer controlled universal tester, used with a 20 kN capacity load cell with standard three point bending fixtures. The size of the specimens, based on EN ISO 14125 was 100 × 10 × 2 mm (length × width × thickness). The test speed was 2 mm/min, and the span length was 80 mm. The temperature was 22.9 °C and the relative humidity was 52%. During the test, force and displacement values were recorded and the bending parameters (σ_{fm}, E_{fm}) were calculated according to the standard.

In the case of the sandwich composite structures the size of the specimens was 120 × 25 × 7.5 and 120 × 25 × 21 mm (length - × width × thickness), the span length was 100 mm and the test speed was 5 mm/min.

3. Results and discussion

3.1. Jute fibre reinforced composites

In order to compare the mechanical properties of the different jute/EP composites, DMTA measurements were carried out at first. The storage modulus (E′) and tanδ curves were recorded in the function of temperature (T). Fig. 1 shows the DMTA curves of the EP composites cured with AR917 and DETDA curing agents.

From the DMTA curves, storage modulus at 0 and 200 °C (before and after the glass transition) and T_g values (based on the tanδ peaks) were determined by the software of the DMTA device. Table 1 shows the determined storage modulus and glass transition temperature (T_g) values.

According to Fig. 1 and Table 1, below T_g GFTE cured with DETDA had higher storage modulus values than the petrol oil based composites (DGEBA, PER, GER), while above T_g and in case of AR917 the tendency was the opposite. As for the T_g values, according to Table 1, GFTE cured with DETDA had only 12 °C lower T_g (167 °C) than the DGEBA based composite, which is an outstanding result for a bio-based EP (e.g. both fatty acid triglyceride [7] and isosorbide based [17] natural sourced EP systems had lower T_g, than DGEBA). In case of AR917 the difference between GFTE and DGEBA was even less, 6 °C. The aliphatic resins, which can be potentially synthesized from renewable materials as well, had much lower T_g, than the aromatic DGEBA or the cycloaliphatic GFTE, consequently in high temperature applications they do not represent a real bio-based alternative. These results are in good agreement with the results of the matrices itself published previously [28].

To compare the mechanical properties of jute/EP composites, tensile and bending tests were carried out. Fig. 2 shows the tensile strength (σ_m), tensile modulus (E_m), bending strength (σ_{fm}) and bending modulus (E_{fm}) of jute/EP composites cured with AR917 and DETDA.

According to the mechanical test results displayed on Fig. 2, in the case of GFTE cured with AR917 the tensile strength and the Young's modulus were significantly lower than in mineral oil based composites. With DETDA however, the DGEBA and the GFTE composites had similar test results. As for the bending properties, with DETDA noteworthy difference between the composites was observed, contrary to the composites cured with AR917. Furthermore, it has to be noted that in each case composites crosslinked with DETDA were weaker than the ones cured with AR917. This phenomenon may be explained by the degradation of jute fibre at 175 °C, necessary to achieve complete curing by DETDA. Namely, at this temperature the bonding agents (lignin, hemicellulose) of the cellulose based fibres can start to degrade already [30]. Typically, the jute fibre has an average tensile strength of 370 MPa, according to Saha et al. [31]. This value is practically in the same order of magnitude as the tensile strength of the applied neat matrices (the average tensile strength of DGEBA is 77.6 MPa [14]). According to these facts, the mechanical properties of the matrices significantly influenced the mechanical properties of jute fibre reinforced composites. With the much stronger CF reinforcement, ($\sigma_m = 5300$ MPa; $E_m = 276$ GPa [32]) this effect can be eliminated.

3.2. Carbon fibre reinforced composites

To investigate the applicability of the sugar based EP monomer in the field of the structural polymer composites, CF reinforcement had been also studied. At first DMTA measurements were carried out. The storage modulus (E′) and tanδ curves were recorded in function of the temperature (T). Fig. 3 shows the DMTA curves composites with AR917 and DETDA curing agents.

From the DMTA curves, storage modulus at 0 and 200 °C (below and above the glass transition temperature) and T_g values (based on the tanδ peaks) were determined by the software of the DMTA device. Table 2 shows the determined storage modulus and T_g values.

According to Fig. 3 and Table 2, under the T_g there was no significant difference between the storage modulus values of GFTE and the reference composites. As for the T_g values, in case of DETDA, the difference between DGEBA and GFTE was higher with CF (23 °C) than with jute fibre reinforcement (12 °C), while with AR917, the difference was just 2 °C. The T_g value of GFTE DETDA composite (161 °C) is still above the results of the usual bio-based EPs, making them appropriate for some high temperature applications as well.

The mechanical properties of the CF/EP composites were compared by tensile and bending tests. Fig. 4 shows the tensile strength (σ_m), tensile modulus (E_m), bending strength (σ_{fm}) and bending modulus (E_{fm}) values of the CF/EP composites cured with AR917 and DETDA.

According to Fig. 4 there was no significant difference between the mechanical properties (tensile strength, tensile modulus, bending strength and bending modulus) of the sugar based and the mineral oil based composites. The GFTE/CF composites had almost in every case (except the bending strength with AR917) better mechanical properties than the DGEBA/CF composites, usually used as benchmark material in aeronautical applications. According to these results, the novel GFTE can be a potential bio-based replacement for the mineral oil based DGEBA EP component.

3.3. Composite sandwich structure

Composite sandwich structures play important role in aeronautical applications, especially in case of indoor elements as e.g.

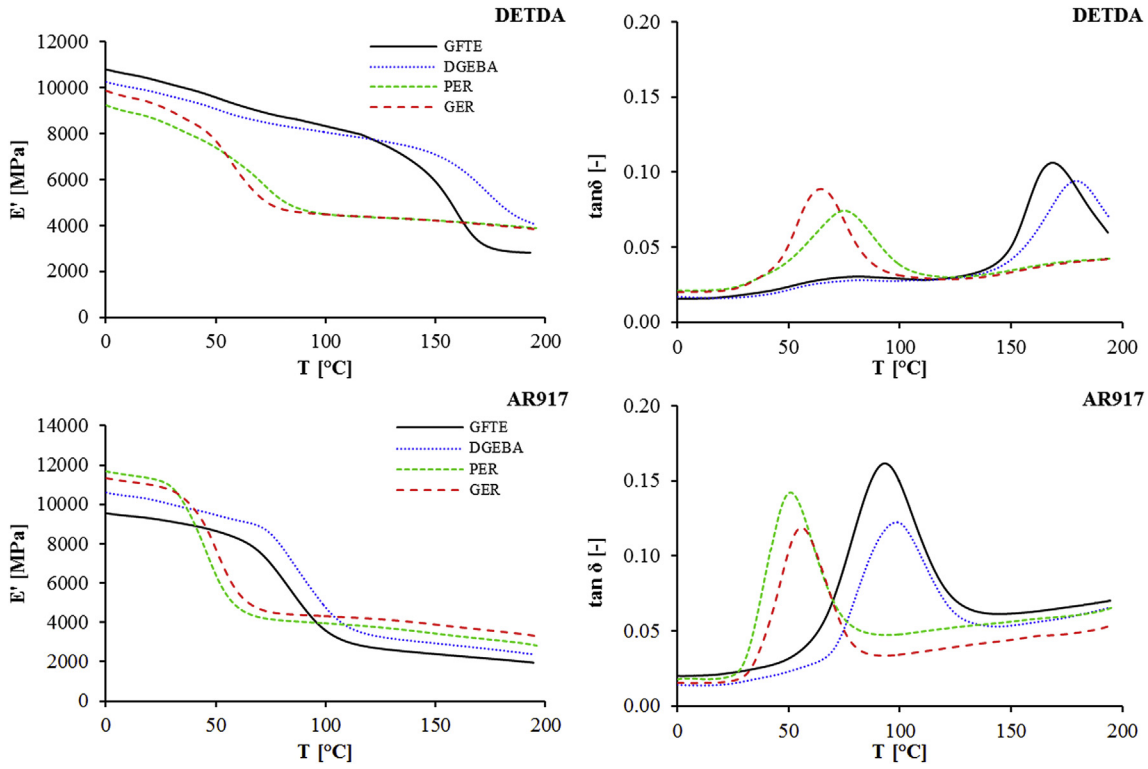

Fig. 1. Storage modulus (E′) and tanδ curves in the function of temperature (T) of jute/EP composites cured with AR917 and DETDA curing agents.

Table 1
Storage modulus (at 0 and 200 °C) and T_g values of jute/EP composites with AR917 and DETDA curing agents.

Base resin curing agent		GFTE		DGEBA		PER		GER	
		DETDA	AR917	DETDA	AR917	DETDA	AR917	DETDA	AR917
Storage modulus (MPa)	0 °C	10806	9551	10257	10603	9242	11682	9874	11338
	200 °C	2812	1951	4075	2371	3885	2820	3846	3303
T_g (°C)		167	93	179	99	75	51	65	56

aircraft floors, ceilings, sidewalls and stowage compartments. Therefore our aim was to compare the properties of sandwich composite structures prepared from a core material, preferred in aeronautical applications, using DGEBA and GFTE as matrices, cured with AR917 curing agent. Jute fibre reinforcement was used to reach as much as possible renewable sourced composite sandwich structures. In the case of the indoor elements the bending strength and modulus are the most important properties; therefore three point bending tests were carried out on the sandwich composites.

According to the test results both in the case of 6.5 and 20 mm thick core, after the bending stress value reached a maximum, it started to decrease due to the failure of the upper composite layer of the sandwich composite structure. After that two different phenomena took place: In the case of the 6.5 mm core, the bending stress decreased further, while with 20 mm core, the bending stress increased until the breaking of the specimen. Two different types of failure occurred: in the case of the 6.5 mm core, when the sandwich structure broke, the specimen stayed together (non-catastrophic failure), while in the case of the 20 mm core, the failure of the specimens was catastrophic. In the case of the 6.5 mm core, the thin core had higher force intermediary and low damping properties, than the 20 mm core. It caused the force decreasing, resulting in non-catastrophic failure at the end of the bending tests. GFTE sandwich composites with 6.5 mm core had significantly better average bending properties than the DGEBA composites (bending strength: GFTE – 55.07 ± 1.20 MPa; DGEBA – 44.22 ± 3.94 MPa, bending modulus: GFTE – 33.93 ± 0.10 GPa; DGEBA – 3.67 ± 0.09 GPa). In the case of the GFTE composites, the average bending strength was 24%, and the average bending modulus was 7% higher than with DGEBA, respectively. With 20 mm core the DGEBA composite had 15% higher bending strength, the modulus values were in the same range (bending strength: GFTE – 13.48 ± 0.36 MPa; DGEBA – 15.90 ± 0.32 MPa, bending modulus: GFTE – 0.68 ± 0.02 GPa; DGEBA – 0.70 ± 0.03 GPa). Based on these results, the bio-based GFTE can replace the DGEBA EP component in the sandwich composite structures, with jute fibre reinforcement, especially in the case of the thinner cores, for example the 6.5 mm one. No interfacial failure between the composite layers and the core material was observed, which indicates a good adhesion between the composite resin system and the core foam.

4. Conclusions

Jute and carbon fibre reinforced composites, as well as jute fibre reinforced sandwich composites with polymethacrylimide foam as core, were prepared using a newly synthesized, renewable glucofuranoside based epoxy resin (GFTE) as polymer matrix. The

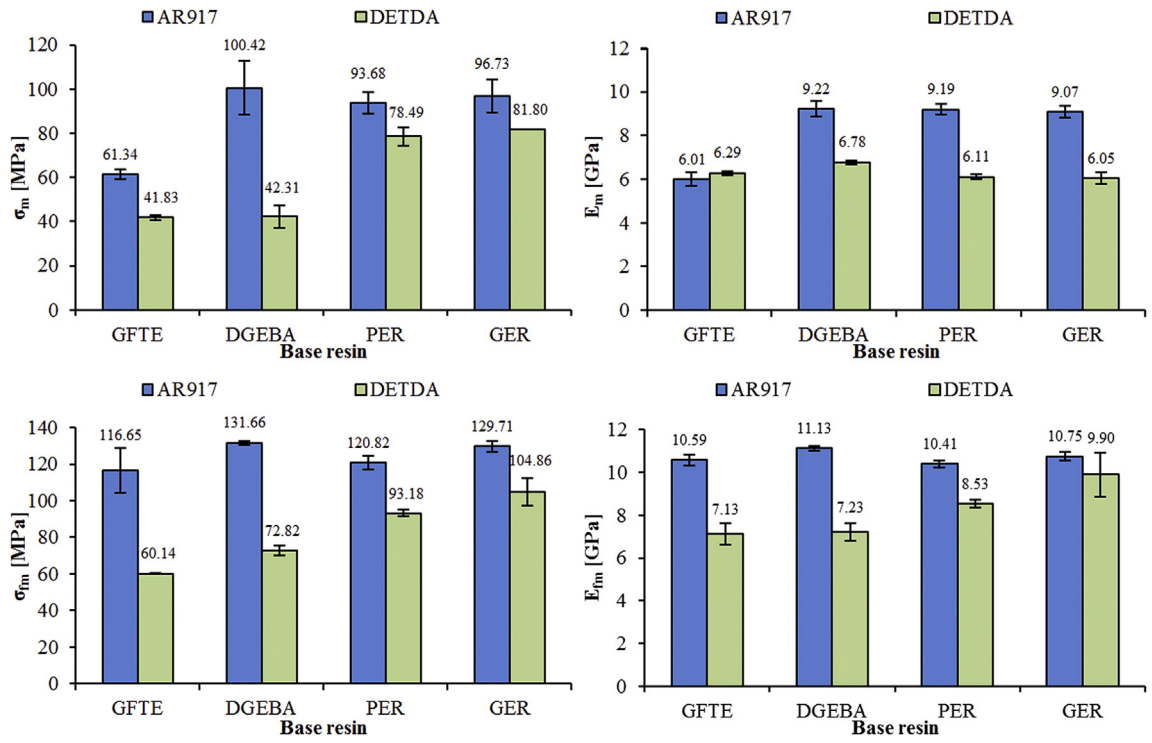

Fig. 2. Tensile strength (σ_m), Young's modulus (E_m), bending strength (σ_{fm}) and bending modulus (E_{fm}) values of jute/EP composites cured with AR917 and DETDA.

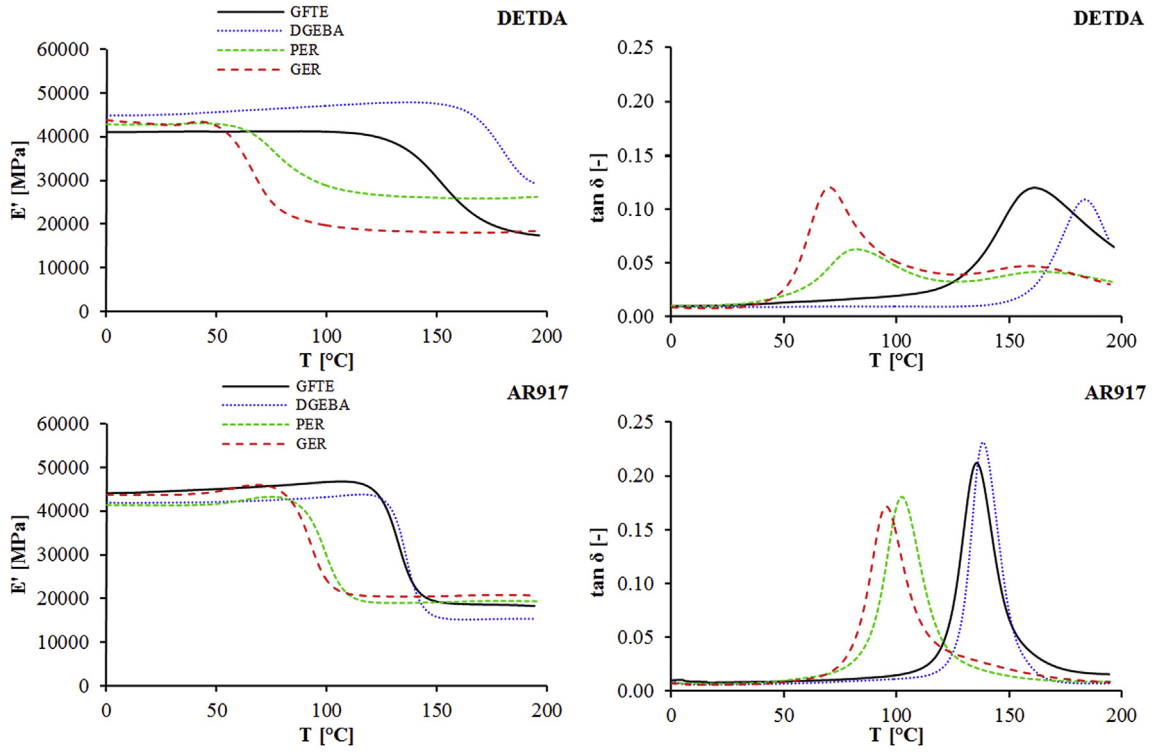

Fig. 3. Storage modulus (E′) and tanδ curves in function of the temperature (T) of the CF/EP composites cured with AR917 and DETDA curing agents.

dynamic mechanical and mechanical properties of the composites were compared to mineral oil based benchmark epoxy resin (DGEBA, PER, GER) composites.

In case of natural jute fibre reinforcement the overall mechanical performance of the GFTE composites was somewhat lower than that of DGEBA composites, with comparable T_g values. The application of the amine type DETDA curing agent, used in high-tech sectors to provide high T_g, lead to degradation of the natural fibres and consequently to lower mechanical properties than in case of anhydride type crosslinking agent.

Table 2
Storage modulus (on 0 and 200 °C) and T_g values of the different CF/EP composites with AR917 and DETDA curing agents.

Base resin curing agent		GFTE		DGEBA		PER		GER	
		DETDA	AR917	DETDA	AR917	DETDA	AR917	DETDA	AR917
Storage modulus (MPa)	0 °C	41038	44065	44869	41919	42833	41392	43786	43770
	200 °C	17416	18308	29345	15360	26229	19358	18409	20706
T_g (°C)		161	136	184	138	82	102	70	95

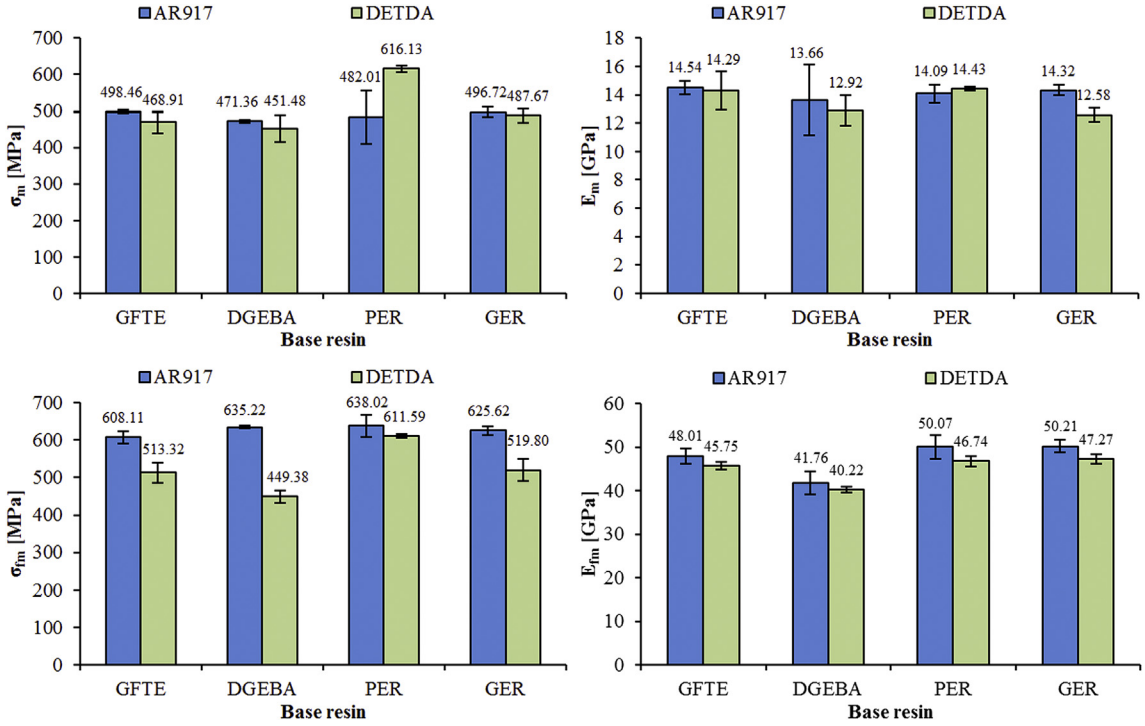

Fig. 4. Tensile strength (σ_m), Young's modulus (E_m), bending strength (σ_{fm}) and bending modulus (E_{fm}) values of CF/EP composites cured with AR917 and DETDA.

In order to eliminate the drawbacks of natural fibres (low tensile strength and thermal stability) and to get an insight into the real performance of GFTE in comparison with DGEBA, composites with a technical carbon fibre were tested. In this case the tensile strength, tensile modulus, bending strength and bending modulus of the GFTE/CF composites was higher than in the case of the DGEBA, and the T_g values of the DGEBA/CF and the GFTE/CF composites were comparable.

For potential aircraft interior flooring applications sandwich structures were prepared with DGEBA and GFTE matrices cured by AR917 curing agent using jute fibre reinforcement and polymethacrylimide foam as core. With 6.5 mm thick core material, the bending strength and modulus of sandwich composite was significantly higher with GFTE than with DGEBA.

These results suggest that the novel glucose-based epoxy monomer can be an alternative to the commonly used mineral oil based diglycidyl-ether of bisphenol-A (DGEBA), even in high temperature applications up to 160 °C using an amine type curing agent approved for aircraft composites.

Acknowledgements

The research leading to these results has received funding from the European Union's Seventh Framework Programme (FP7/2007–2013) for the Clean Sky Joint Technology Initiative under grant agreement no 298090 (Topic manager: Dassault Aviation). This work is connected to the scientific program of the "Development of quality-oriented and harmonized R + D + I strategy and functional model at BME" and "Talent care and cultivation in the scientific workshops of BME" project. This project is supported by New Széchenyi Plan (Project ID: TÁMOP-4.2.1/B-09/1/KMR-2010-0002), by NFÜ EU_BONUS_12-1-2012-0026, by TÁMOP – 4.2.2.B-10/1–2010-0009. A. Toldy and G. Szebényi acknowledge the financial support received through János Bolyai Scholarship of the Hungarian Academy of Science. The authors would like to thank Beáta Szolnoki and Zsolt Rapi for the synthesis of glucose based epoxy monomer, Tibor Czigány and József Karger-Kocsis for useful comments, from Budapest University of Technology; and Bertrand Soulezelle at Dassault Aviation for advices concerning the preparation of sandwich composites for aircraft interior flooring.

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