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Dedicatory

To **Prof. Stephen W. Tsai**, that has devoted his career to research in composite materials. One of the most renowned composite engineers, he is known for the Tsai-Hill failure criterion, lamination parameters, the Tsai-Wu failure criterion and strength ratios, the Halpin-Tsai micromechanics model, trace, master ply, omni envelopes and unit circle failure criterion, among others.

Prof. Tsai has thus far published nine books (not including conference series): (1) Introduction to composite materials, (2) Composite Materials: design and applications, (3) Composites design, (4) Structural behaviour of composite materials, (5) Analyses of composite structures, (6) Strength & life of composites, (7) Theory of composites design, (8) Composite Materials Design and Testing, and (9) Design of Composite Laminates.

He is still active as an emeritus professor at Stanford University, and his lifelong talent and mastery in the field have recently led to the development of the Double-Double composite manufacturing and design technique, the theme of his new book launched this year (2022).



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About the 6th BCCM



The oldest train in operation in Brazil was inaugurated in 1881 by Dom Pedro II and it travels 12 km between Tiradentes and São João del-Rei.

6th Brazilian Conference on Composite Materials

The 6th BCCM was attended by over 300 participants from 14 countries. More than 100 articles have been published and presented orally, with authors from more than 60 research, teaching and technology institutions. The 6th BCCM was held from 14th to 18th August 2022, in *Tiradentes*, a charming and warmhearted historical city famous for its gastronomy and architecture. *Tiradentes* was founded in 1718, named after *Joaquim José da Silva Xavier*, the *"Tiradentes"*, patron of the Brazilian Republic.

Since its first edition in 2012, the Conference congregates Brazilian and worldwide students, professors, and researchers from academia and industry to promote the recent developments and discuss the challenges in composite materials in Brazil and abroad. The range of topics is comprehensive, including Damage and Fracture; Simulation in Composites; Adaptive Composites; Durability and Ageing; Mechanical and Physicochemical Properties of composites; Nanocomposites; Recycling and reuse of composite materials; Experimental Techniques; Lignocellulosic Composites; Processing and Manufacturing of composites; Health Monitoring in composite structures; Multi-Scale Modelling; Industrial Applications.



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Behind the 6th BCCM

The 6th Brazilian Conference on Composite Materials was co-organised by the Federal University of São João del-Rei (UFSJ), the Federal University of Minas Gerais (UFMG), and the Centre for Innovation and Technology in Composite Materials (CIT^eC).

Ranked among Brazil's top Institutions of Higher Education and located a short distance from the state capital city of *Belo Horizonte*, **UFSJ** offers a wide selection of over 50 graduate and undergraduate courses in areas such as Health Sciences, Arts and Humanities, Music, Social Sciences, Engineering and Technology, and Basic Sciences. Courses are taught on six different campuses, three of them located in *São João del-Rei* and the other three in the towns of *Divinópolis, Ouro Branco*, and *Sete Lagoas*. The University offers a stimulating environment for academic work, and at the same time, situated as it is in the historical heart of the state of *Minas Gerais*, it gives access to a range of cultural activities and attractions.

Placed in the southeast of Brazil, the most industrialized region of the country, **UFMG**, a free-of-charge public educational institution, is the oldest university in the state of *Minas Gerais*. It was founded in September 7th 1927 by the name of University of Minas Gerais (UMG). Nearly one hundred years later the institution is the national leader when it comes to education, university extension, culture, scientific research and patent generation in several fields of knowledge.

CIT^e**C** is a research group created in 2011 at UFSJ for the development and characterisation of laminate, particulate, hybrid and structural composites consisted of polymer and ceramic matrices for aeronautical, automotive, leisure, civil construction and orthopaedic applications. One of the pillars of the group is the research of sustainable materials through the use of natural fibres and recycled wastes. The group stands out for the use of statistical techniques in the evaluation of composite materials, counting on a significant number of articles published in journals of high impact. National and international collaborators have been contributing to the effective growing of the group. Visiting fellows, MSc and PhD students have been also a great motivation for our research team.

Conference Schedule

Time	Sunday ^{14th}	Monday ^{15th}
07:30		Register
08:00		Opening ceremony
08:30		Plenary 1 (Ramesh Talreja)
09:20		Presentation 1 (Room 1-3)
10:40		Coffee-Break
11:05		Presentation 2 (Room 1-3)
12:25	Register	Lunch
14:15	Short-courses: part I	Plenary 2 (Steven Tsai)
15:05	(Adriano Koga; Sérgio Pezzin)	Presentation 3 (Room 1-3)
16:25	Coffee-Break	Coffee-Break
16:50	Short-courses: part II	Presentation 4
17:40	(Adriano Koga; Sérgio Pezzin)	(Room 1-3)

Time	Tuesday ^{16th}	Wednesday ^{17th}	Thursday ^{18th}
08:30	Plenary 3 (Fabrizio Scarpa)	Plenary 5 (Pedro Camanho)	Technical Lectures (Anton Paar; HEXCEL)
09:20	Presentation 5 (Room 1-3)	Presentation 7 (Room 1-3)	Presentation 11 (Room 1-3)
10:40	Coffee-Break	Coffee-Break	Closing ceremony
11:05	Presentation 6 (Room 1-3)	Presentation 8 (Room 1-3)	
12:25	Lunch	Lunch	
13:55	Plenary 4 (Eloi Figueiredo)	Plenary 6 (Mauricio Donadon)	
14:45	Short-courses: part I (Sandro Amico; Flamínio Levy Neto; Gerson Marinucci)	Presentation 9 (Room 1-3)	
16:05	Coffee-Break	Coffee-Break	
16:30 17:50	Short-courses: part II (Sandro Amico; Flamínio Levy Neto; Gerson Marinucci)	Presentation 10 (Room 1-3)	
20:00		Dinner	

Keynote Speakers – Plenaries

Three decades of pattern recognition for structural health monitoring of bridges



Prof. Elói Figueiredo Lusófona University Portugal

Strength prediction of composite laminates

under uncertainties using theory-guided

machine learning

Mechanical metamaterials and biobased composites: new trends for sustainable high-performance structures



Prof. Fabrizio Scarpa University Of Bristol United Kingdom

Failure analysis of composite materials towards cost/performance trade-off



Prof. Pedro Camanho University of Porto Portugal



Prof. Ramesh Talreja Texas A&M University USA

The analytic foundation of double-double: DD concepts & operation



Prof. Stephen Tsai Stanford University USA

A decohesive interface element for static and fatigue induce damage predictions in adhesively bonded joints under variable loads and debonding mode ratios



Prof. Mauricio Donadon Aeronautics Institute of Technology Brazil

Short Courses

Finite Element Method (FEM)



Adriano Koga Altair 🛆

Fundamentals of polymeric matrix composites



Prof. Gerson Marinucci Nuclear and Energy Research Institute Analysis of laminated composite beams



Prof. Flamínio Levy Neto University of Brasília

Introduction to composite manufacturing



Prof. Sandro Amico Federal University of Rio Grande do Sul

Self-healing composites



Prof. Sérgio Pezzin Santa Catarina State University

Technical Lectures

Characterisation of mechanical properties in composite materials



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About published articles

The 6th BCCM featured the publication of 119 articles, and all work presentations were performed orally, distributed in 3 rooms with 11 presentation sessions each. The presenter's name is underlined in the manuscript header.

In this book section, the reader will find a list with all participating institutions (authors' affiliation), a summary with all the titles of published articles, and the schedule for the oral presentations performed at the conference.



Serra de São José, with a maximum altitude of approximately 1300 m, is located between the municipalities of Tiradentes, São João del-Rei, Santa Cruz de Minas, Prados and Coronel Xavier Chaves.

Participating institutions (author's affiliation)

Algeria 🔮

• University of Guelma

Austria 🗲

• FRACTURE ANALYTICS

Brazil 📀

- Aeronautics Institute of Technology
- Amazonas State University
- Braskem S.A.
- Brazilian Army Technological Center
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- Federal Center for Technological Education of Minas Gerais
- Federal Center of Technological Education Celso S. da Fonseca
- Federal Fluminense Institute
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- Technological University of Habana

Germany

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- Toray Advanced Composites
- University of Twente

United Kingdom 🏶

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Rheology behavior of epoxy/carbon nanoparticles suspension

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Abstract: The use of nanoparticles as additives to obtain different functionalities in resins such as epoxy has recently attracted much attention. In this work, the rheological behavior of suspensions of graphene nanoparticles (GN) and carbon nanotubes (CNT) (0.05-2 wt.%) were investigated. Graphene oxide (GO) and carbon dots (CQD) are also being investigated, being reactive species that could act as compatibilizers. The suspensions were produced by mechanical stirring (30 min) followed by ultrasonication (30 min). The Bagley diagram was constructed with the solubility parameters (δ_i) of the nanofillers, and it was noted that the GO is more compatible with the hardener (TETA), while CNT and CQD with the resin. CNT system showed rheology percolation with a content above 0.2 wt.%, whereas with NG no percolation was observed up to 2 wt.%.

1. Introduction

The use of nanoparticles as additives to obtain different functionalities in resins such as epoxy has recently attracted much attention, for instance, coatings with microbial action using silver nanoparticles [1], adhesives using graphene (GN) [2], conductive materials with carbon nanotubes (CNT) [3], and anti-counterfeiting materials such as carbon quantum dots (CQDs) [4]. Nonetheless, the specific functionalities of the resin involve many steps, such as obtaining a stable suspension so that its properties are little affected during processing.

The low solubility is a significant obstacle for the effective dispersion in liquids and resins of carbonic nanoparticles such as GN. This is related to the van der Waals forces that promote their agglomeration and the number of polar groups in their structure, resulting in hydrogen bonds, yielding stable suspensions [5]. An

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alternative is to use surfactants to aid in dispersion and reduce re-agglomeration [6]. However, this additive can be a defect former in applications such as structural composites. Other strategies such as the inclusion of surface groups can be employed, but this requires further processing steps and brings difficulties in scaling-up. These observations are also noted for CNT. Besides, the rheological percolation limit for CNT is lower compared to GN.

Nanoparticles such as graphene oxide (GO) and carbon quantum dots (CQDs) have to surround chemical groups with active hydrogen and are very soluble in polar solvents and resins such as epoxy. This is because, in their structure, there are oxygenated and nitrogenated chemical groups with active hydrogen [7]. Thus, these nanoparticles can be used as a compatibilizer and other carbonic particles with lower polarity, such as GN and CNT. However, the rheological properties of the resin and its hardener must be little affected to prevent composite processing difficulties and to guarantee its applicability.

This work investigates the epoxy resin (DGEBA/TETA) flow behavior using different carbonic fillers (GN and CNT), as well as the use of carbonic particles such as GO and CQDs as compatibilizers.

2. Experimental

An epoxy resin (DGBEA) with an average molecular weight of 750 g.mol⁻¹ (GPC measurements, THF, and polystyrene standards) with code AR260 was acquired from e-composites (Brazil). The triethylenetetramine (TETA) with code HY951 was purchased from Hustman of Brazil. Graphene nanoplatelets with an average thickness of 5 – 10 nm in varying sizes up to 25 μ m under and multi-walled CNT (diameter ~140 nm, length ~7 μ m) were purchased from Strem Chemicals, Inc. USA. GO was obtained through the modified Hummers method, according to the literature. CQDs were obtained by microwave-assisted reactions and slow pyrolysis, described in [7-8].

Epoxy and hardener suspensions were prepared using 0–2 wt.% nanoparticles. The nanoparticles were first mechanically stirred separately in the resin and hardener at a speed of 13000 rpm (Ultra-Turrax T 18) for 30 min in an ice bath. After that, they were sonicated for 30 min in a Sonifier® Model W-450D (amplitude: 60%; pulse on/off, 60 s/60 s) cooled with ice bath. The stability of the suspensions was assessed with visual analysis in graduated cylinders for a minimum time of 7 days. The effect of the particles as compatibilizers was evaluated in the sample with 0.2 wt.% in which half of the GN or CNT was replaced by GO or CQDs. Up-and-down flow curves were obtained in an Anton Paar MCR 101 rheometer using 25 mm parallel plate geometry at 25°C for shear rates of 0.1-1000 s⁻¹.

3. Results and Discussions

To evaluate the solubility of nanoparticles and resin, 2D plots of solubility parameters were employed (Bagley plots) (Fig. 1). This methodology assumes that the effects of the dispersion (δ_d) and polar (δ_p) components of the solubility parameter show close similarity, while the effect of the hydrogen-bonding (δ_h) component is from an entirely different nature. Dispersive, polar, and hydrogen contributions were computed using literature data [9-12]. In the Bagley plot with epoxy resin in the circle's center, the solubility of the nanoparticles in the resin system can be evaluated. As can be seen, GO is more miscible with the hardener $D_{12} = 9.65$ MPa^{1/2} compared to GN, whereas CNT (6.29 MPa^{1/2}) and CQDs (6.71 MPa^{1/2}) are more miscible with pure resin. GN is the nanoparticle with lower miscibility with both resin and hardener.

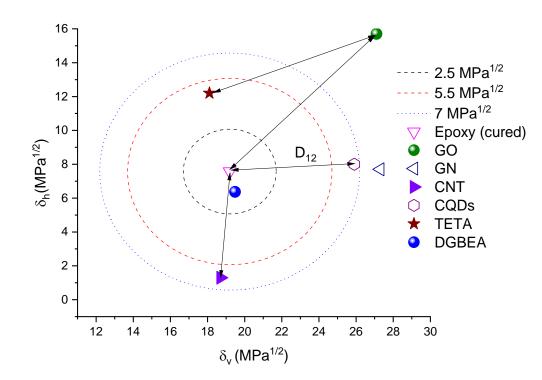


Fig. 1. Bagley plots for epoxy, hardener and nanoparticles. The dotted circles indicate D_{12} equals to 2.5, 5.5 and 7.0 MPa^{1/2}.

Fig. 2 a-b) show the epoxy resin up-and-down flow curves. No sample showed a significant hysteresis loop. For the system with GN, all suspensions showed a Newtonian behavior, while for those with CNT, the non-Newtonian viscosity behavior depended on the amount of CNT. The Herschel-Bukley model was used and fitted to the systems, with $R^2 > 0.999$. Above 0.2 wt.%, a more significant deviation from the Newtonian behavior is noticed due to the formation of a percolated network, in which the particles significantly increase viscosity of the system. Regarding the TETA/GN and TETA/CNT suspensions, a similar behavior was observed, i.e., a Newtonian behavior with GN and a percolation threshold with 0.2 wt.% of CNT. Addition of CNTs to a Newtonian suspension (resin) increased the apparent shear viscosity at low shear rate and, as the shear rate increased, the viscosity decreased asymptotically to that of the suspension. This type of behavior is known as shear-thinning, and is caused by many factors, such as chemical modification of particles and others. In the current work, it may occur due to a hierarchical structure of aggregated CNT structures [13].

Table 1 shows the fitted rheological data using the Herschel-Bukley model. The τ_0 term is nearly zero, and a rheological behavior of a power-law fluid ($\tau = K\dot{\gamma}^n$) was considered. The systems with GN deviate very little from the Newtonian behavior, as *n* is very close to 1. In addition, the value of the consistency index, *K* tends to increase when CQDs and GO are added, which indicates an increase in fluid consistency. A reduction in this parameter for suspensions with CNT is seen, which is similar to what was reported by Ma et al [14] when comparing the rheological behavior of CNT suspensions with and without treatment. This indicates that CQDs can essentially cause the CNT to behave like short fibers that rotate and align under shear flow.

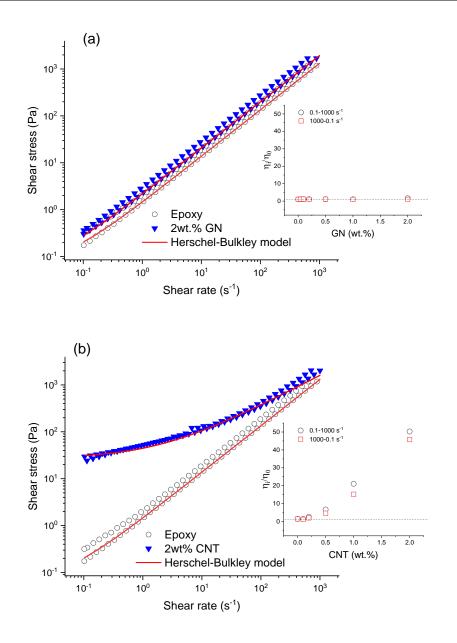


Fig. 2. Up-and-down flow curves of epoxy resin with GN (a) and with CNT (b). Red line represents the Herschel-Bulkey model. Insert refers to the relative viscosity at 10^{-1} s⁻¹ as a function of nanoparticle content.

Sample	$ au_0$ (Pa)	K (Pa.sn)	n	R ²
Ероху	-	1.368	0.996	0.9999
0.2 GN	-	1.336	0.996	0.9999
0.1 GN + 0.1 CQDs	-	1.414	0.993	0.9999
0.1 GN + 0.1 GO	-	1.656	0.959	0.9991
0.2 CNT	-	2.169	0.929	0.9957
0.1 CNT + 0.1 CQDs	-	1.323	0.992	0.9999

Table 1. Rheological fitted parameters of compatibilized suspensions.

4. Conclusions

Epoxy and TETA suspensions with different carbonic particles were prepared and their rheological behavior was investigated. The solubility parameters contributed to understanding which nanoparticle is more soluble in the resin and the hardener. The system with GN did not present rheological percolation, while for CNT percolation occurred above 0.2 wt.%. This approach showed the possibility of using nanoparticles such as GO and CQDs as GN compatibilizers in epoxy systems due to the presence of specific functional groups (-OH, -COOH and –NH₂) in the nanoparticles.

Declaration of Competing Interest

The authors declare no conflict of interest.

Credit author statement

J A. Cruz: Conceptualization; Methodology; Formal Analysis. L Paim: Conceptualization; Methodology; Formal Analysis. R Joffe: Writing – review. P Fernberg: Writing – review. S Amico: Supervision; Writing – review. O Bianchi: Conceptualization; Writing – original draft; Resources; Supervision

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