

RGC Ref. No.: UGC/FDS24/E07/20 <p>(please insert ref. above)</p>
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**RESEARCH GRANTS COUNCIL
COMPETITIVE RESEARCH FUNDING SCHEMES FOR
THE LOCAL SELF-FINANCING DEGREE SECTOR**

FACULTY DEVELOPMENT SCHEME (FDS)

Completion Report
(for completed projects only)

<p><u>Submission Deadlines:</u></p> <ol style="list-style-type: none"> 1. Auditor's report with unspent balance, if any: within <u>six</u> months of the approved project completion date. 2. Completion report: within <u>12</u> months of the approved project completion date.
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Part A: The Project and Investigator(s)

1. Project Title

Development of Plant Fiber-Reinforced Polymer Composites (PFRPCs) with Enhanced Mechanical and Flame-Retardant Properties

發展提高力學及阻燃性能的植物纖維增強聚合物複合材料

2. Investigator(s) and Academic Department(s) / Unit(s) Involved

Research Team	Name / Post	Unit / Department / Institution
Principal Investigator	Dr NG Sun-pui / Associate Division Head and Principal Lecturer	Division of Science, Engineering and Health Studies / School of Professional Education and Executive Development, The Hong Kong Polytechnic University (PolyU SPEED)
Co-Investigator(s)	Prof LEUNG Chun-wah / Professor	Director's Office / PolyU SPEED
Co-Investigator(s)	Prof KAN Chi-wai / Professor	School of Fashion and Textiles / The Hong Kong Polytechnic University
Co-Investigator(s)	Dr WOO Eric Kin-sang / Former Principal Lecturer	N/A

Research Team	Name / Post	Unit / Department / Institution
Co-Investigator(s)	Dr CHIU Wang-kin / Senior Lecturer	Division of Science, Engineering and Health Studies / PolyU SPEED
Others	Dr CHAN Wing Yu / Research Assistant	General Office / PolyU SPEED (Funded by FDS)
	Miss MAK Yuen Sum / Research Assistant	General Office / PolyU SPEED (Funded by FDS)

3. Project Duration

	Original	Revised	Date of RGC / Institution Approval (must be quoted)
Project Start Date	01/01/2021	N/A	N/A
Project Completion Date	31/12/2023	N/A	N/A
Duration (<i>in month</i>)	36 months	N/A	N/A
Deadline for Submission of Completion Report	31/12/2024	N/A	N/A

- 4.4 Please attach photo(s) of acknowledgement of RGC-funded facilities / equipment.
N/A

Part B: The Final Report

5. Project Objectives

5.1 Objectives as per original application

1. To develop a thorough database for the physical and morphological properties, and mechanical performance of various lignocellulosic fibers (e.g. sisal, coconut, bamboo, fique, hemp, flax, jute and ramie) as well as matrix materials (e.g. epoxy, polyester and vinyl ester) and the corresponding chemical treatments for enhancing the mechanical and flame-retardant properties from literature. These searched findings are used as the baseline properties for comparison purpose and material selection for further experiments in this project.
2. To prepare the matrix material samples by three selected resins with micro-GO particles in different sizes and weight percentages. Standard tensile test and flammability test will be performed to determine the resins' stiffness, strength and flame retardancy. Hence, the optimum GO particle size and amount in weight for each resin can be determined.
3. To determine the effects of APP treatment with different process parameters including polymerizing gas flow rate, discharge power, and duration of plasma exposure on the corresponding changes of plant fiber surface properties. Tensile test, single fiber pull-out test and flammability test will also be performed to determine the tensile strength, interfacial shear strength with the resins mixed with GO particles and flame retardancy of selected plant fibers after APP treatment.
4. To fabricate PFRPC specimens for tensile test and flammability test and verify the enhancement of mechanical and flame-retardant properties.

5.2 Revised objectives

Date of approval from the RGC: N/A

Reasons for the change: N/A

5.3 Realisation of the objectives

(Maximum 1 page; please state how and to what extent the project objectives have been achieved; give reasons for under-achievements and outline attempts to overcome problems, if any)

For the first objective, a thorough database was established by the screened PFRPCs-related data from a systematic literature search in seven electronic databases including Materials Research Database, Comprehensive Composite Materials, Engineered Materials Abstracts, ProQuest Dissertations & Theses, ScienceDirect, Scopus and SpringerLink. Journal papers, conference papers and dissertations were selected for full-text review according to the following inclusion criteria: (1) studies evaluating the physical and morphological properties, and mechanical performance of various lignocellulosic fibers as well as matrix materials; (2) addition of fillers or treatment methods for enhancing the mechanical and flame-retardant properties; and (3) the corresponding fabrication and testing methods for the resulting plant fiber-reinforced polymer composites (PFRPCs).

For the second objective, three types of matrix materials, epoxy, polyester and ethylene-vinyl acetate resins, were used to prepare test specimens with different contents of micro-size graphene oxide (GO) particles made by Hummers method. According to the database obtained after achieving the first objective, the three selected particle sizes were 1 μm , 5 μm and 10 μm while the three selected weight percentages were 1%, 3% and 5%. As it was noticed from the literature database that phosphorus compound is a good flame retardant, the Crystal Green® granular phosphorus fertilizer (CG particles) in weight percentages of 15%, 20%, 25% and 30% were also used to mix with the resin materials for trials. All the specimens were fabricated and tested according to the standard tensile test method ASTM D3039 in order to determine the mechanical properties including the tensile stiffness and strength. In addition, flammability tests were also performed according to the UL-94 Vertical Burn Test. From the experimental results of these two standard tests, the optimal size and amount in weight of GO particles and CG granules for the resin with the best mechanical and flame-retardant performances were determined.

For the third objective, the effects of atmospheric pressure plasma (APP) treatment on three types of plant fibers, namely flax, jute and ramie in woven fabric form, were studied. To prepare the fabric specimens with different plasma exposure conditions, they were treated with different process parameters, i.e. plasma head distances, treatment speeds and two different types of gases (dry air and argon). PFRPC laminates were made by bonding 3 plies of APP treated fabrics using the selected resin with optimal content of GO or CG particles with the aids of a hydraulic press machine. Control PFRPC laminates were also prepared by using 3 plies of untreated fabrics. The tensile and flame-retardant properties of the PFRPC laminate specimens were evaluated by the aforementioned tensile and flammability tests, while the bonding strength between the selected plant fiber and resin materials were measured by the interlaminar shear strength test. Moreover, the topographical and chemical properties were obtained by scanning electron microscopy (SEM) and fourier-transform infrared spectroscopy (FTIR).

For the fourth objective, PFRPC laminate specimens reinforced with the chosen plant fiber were made up of 5 and 7 plies of fabrics and all pieces of fabrics were APP treated using the optimal process parameters. The same tensile and flammability tests were conducted and the enhancements of mechanical and flame-retardant properties were verified.

5.4 Summary of objectives addressed to date

Objectives (as per 5.1/5.2 above)	Addressed (please tick)	Percentage Achieved (please estimate)
1. To develop a thorough database for the physical and morphological properties, and mechanical performance of various lignocellulosic fibers (e.g. sisal, coconut, bamboo, fique, hemp, flax, jute and ramie) as well as matrix materials (e.g. epoxy, polyester and vinyl ester) and the corresponding chemical treatments for enhancing the mechanical and flame-retardant properties from literature. These searched findings are used as the baseline properties for comparison purpose and material selection for further experiments in this project.	✓	100%
2. To prepare the matrix material samples by three selected resins with micro-GO particles in different sizes and weight percentages. Standard tensile test and flammability test will be performed to determine the resins' stiffness, strength and flame retardancy. Hence, the optimum GO particle size and amount in weight for each resin can be determined.	✓	100%
3. To determine the effects of APP treatment with different process parameters including polymerizing gas flow rate, discharge power, and duration of plasma exposure on the corresponding changes of plant fiber surface properties. Tensile test, single fiber pull-out test and flammability test will also be performed to determine the tensile strength, interfacial shear strength with the resins mixed with GO particles and flame retardancy of selected plant fibers after APP treatment.	✓	100%
4. To fabricate PFRPC specimens for tensile test and flammability test and verify the enhancement of mechanical and flame-retardant properties.	✓	100%

6. Research Outcome

6.1 Major findings and research outcome

(Maximum 1 page; please make reference to Part C where necessary)

From the tensile test results of the resin specimens with GO or CG particles, it was found that epoxy resin exhibited the highest tensile stiffness and strength when compared with the other resins. The optimal size and weight percentage of GO particles dispersed in epoxy resin (GO/epoxy) were 1 μm and 3% respectively. The stiffness and strength of GO/epoxy specimens increased when the GO content increased from 1 wt% to 3 wt% but dropped when it reached 5 wt%. The stiffness and strength of the 3 wt% GO/epoxy were 794.6 MPa and 49.3 MPa respectively. For the CG particles, the stiffness and strength of CG/epoxy also showed an upward and downward trend as the CG content increased, with 25% being the optimal weight percentage. The stiffness and strength of the 25% CG/epoxy were 749.9 MPa and 36.4 MPa respectively.

For the flammability test results, the rating of pure epoxy and 1 wt% GO/epoxy in the UL-94 vertical burn test was “no rating”, as they were unable to self-extinguish. For the 3 wt% and 5 wt% GO/epoxy materials, their burning time after initial flame application was less than 10 seconds and their afterglow time periods following the second ignition were between 30 to 60 seconds (V-1 rating) and less than 30 seconds (V-0 rating) respectively. In other words, GO/epoxy achieved the best flame-retardant performance (V-0 rating) when the GO content reached 5 wt%. While for CG particles, the ratings of 15 wt%, 20 wt%, 25 wt% and 30 wt% CG/epoxy materials were “no rating”, V-1, V-0 and V-0 respectively. These ratings imply that CG/epoxy achieved the best flame-retardant performance when the CG content reached 25 wt%. When considering both of the mechanical and flame-retardant properties of GO/epoxy and CG/epoxy, it is advantageous to employ 25 wt% CG/epoxy as the matrix material for fabricating the PFRPC laminates in the subsequent stages of the current research project.

Based on the tensile and interlaminar shear strength test results of the PFRPC laminates reinforced by 3 plies of untreated and APP-treated plant fiber woven fabrics, it was found that flax fiber exhibited the highest mechanical properties. The optimal process parameters for the APP treatment were the plasma head distance of 5 cm and plasma head speed of 25 mm/s. By comparing the tensile stiffness of untreated flax/epoxy laminate (0.89 GPa), the APP-treated laminates had an increased stiffness by 121.29% using dry air (1.97 MPa) and 96.58% using argon gas (1.75 MPa). Moreover, the ultimate tensile strength of untreated flax/epoxy laminate was 56.52 MPa and that of the APP-treated laminates increased by 5.95% and 2.95% for dry air (59.89 MPa) and argon gas (58.19 MPa) respectively. When comparing the bond strength between fiber and matrix materials in terms of interlaminar shear strength of untreated flax/epoxy laminate (5.98MPa), the APP-treated laminates had an increased interlaminar shear strength by 40.47% (8.4 MPa) and 18.23% (7.07 MPa) using argon gas and dry air respectively. From the SEM micrographs, it was observed that the fractured specimens had more epoxy resin attached to the broken treated fibers and more striation marks left on the epoxy resin due to fiber sliding. These observations were in line with the results of FTIR Spectroscopy. Additional C=O, N-O and C-O compounds were formed on the APP-treated flax fiber. For the flax/epoxy laminates reinforced by 5 plies and 7 plies of APP-treated fabrics. Comparable enhancements in tensile stiffnesses (5-ply: 2.09 GPa; 7-ply: 2.02 GPa) and ultimate tensile strengths (5-ply: 62.88 MPa; 7-ply: 61.09 MPa) were obtained and all specimens had the V-0 rating in the vertical burn tests.

Our findings were published in four international conference papers and will be published in two peer-reviewed journal articles. The details of these papers can be found in Part C of this report.

6.2 Potential for further development of the research and the proposed course of action (*Maximum half a page*)

The newly developed plant fiber-reinforced polymer composites (PFRPCs) exhibit good mechanical and flame-retardant performances, and future research should explore novel properties, such as sound absorption, which are required in many engineering applications. Polyurethane foam is the most prevalent type of sound-absorbing material prevailing on the market, but its high flammability is the main drawback. To this end, a potential research development is to combine low-density porous aerogel particles (e.g., silica and PVA) in the resin of a PFRPC laminate to improve its sound absorption ability while retaining mechanical and flame-retardant properties.

To implement this proposed research, a designated type of aerogel particle will be dispersed in the solvent by using an ultrasonic bath before being mixed into the epoxy resin with the flame-retardant particles. The epoxy mixture will then be utilized to fabricate a new plant fiber-reinforced polymer laminate. In addition to the mechanical tensile and flammability testing, sound absorption tests employing an impedance tube and a reverberation chamber will also be performed to optimize the ratio of each component in the resin based on various performance criteria.

7. Layman's Summary

(*Describe in layman's language the nature, significance and value of the research project, in no more than 200 words*)

Fiber-reinforced polymer composites (FRPCs) have been used tremendously in various engineering applications, such as medical equipment, civil infrastructure, automobiles, aircrafts, aerospace, sporting goods and consumer products etc. Carbon, glass and aramid fibers are commonly used in composites but the raw materials for making these fibers are limited, and the recyclability processes for these fibers are costly and inefficient. As a result, the trend towards the development of “green” fibers as the reinforcement of composites is growing rapidly. Despite of that, the interfacial bonding between the fiber and resin in the plant fiber reinforced composites is relatively weak. Moreover, plant fibers are flammable and release more heat than traditional reinforcement fibers in composites, making them more hazardous to users in case of fire incidents. To address these issues, the current research project developed a new composite material made of APP-treated flax fiber and phosphorus-containing epoxy resin, which has significantly improved mechanical and flame-retardant properties. Not only are the reinforcement fibers and phosphorus particles in the resin biodegradable, but the APP treatment is also an eco-friendly fabrication method.

Part C: Research Output**8. Peer-Reviewed Journal Publication(s) Arising Directly From This Research Project**

(Please attach a copy of the publication and/or the letter of acceptance if not yet submitted in the previous progress report(s). All listed publications must acknowledge RGC's funding support by quoting the specific grant reference.)

The Latest Status of Publications				Author(s) (denote the corresponding author with an asterisk*)	Title and Journal / Book (with the volume, pages and other necessary publishing details specified)	Submitted to RGC (indicate the year ending of the relevant progress report)	Attached to this Report (Yes or No)	Acknowledged the Support of RGC (Yes or No)	Accessible from the Institutional Repository (Yes or No)
Year of Publication	Year of Acceptance (For paper accepted but not yet published)	Under Review	Under Preparation (optional)						
N/A	N/A	✓	N/A	CHAN Wing-Yu, NG Sun-pui*, KAN Chi-wai, LEUNG Chun-wah & CHIU Wang-kin	Effect of Plasma Surface Activation on Bond Strength and Mechanical Performance of Flax/Epoxy Composites Submitted to "Polymer Testing"	No	Yes (Appendix 1)	Yes	No
N/A	N/A	N/A	✓	NG Sun-pui*, CHAN Wing-Yu, LEUNG Chun-wah, KAN Chi-wai & CHIU Wang-kin	Flame-retardant Flax-reinforced Composites using Phosphorus-containing Epoxy Resin Will be submitted to "Composites Part B: Engineering"	No	No [Under Preparation]	Yes (Authors will acknowledge the support of RGC in the manuscript)	No

9. Recognized International Conference(s) In Which Paper(s) Related To This Research Project Was / Were Delivered

(Please attach a copy of each conference abstract)

Month / Year / Place	Title	Conference Name	Submitted to RGC <i>(indicate the year ending of the relevant progress report)</i>	Attached to this Report <i>(Yes or No)</i>	Acknowledged the Support of RGC <i>(Yes or No)</i>	Accessible from the Institutional Repository <i>(Yes or No)</i>
Aug 2022 / Hong Kong	Effect of Plasma Surface Activation on Bond Strength of Flax/Epoxy Composites	International Conference on Advances in Design, Materials and Manufacturing Technologies 2022	No	Yes (Appendix 2)	Yes	Yes
Aug 2022 / Hong Kong	Improved Fire-retardant Performance of Epoxy Resin Composites using Organic Fibre Filler	International Conference on Advances in Design, Materials and Manufacturing Technologies 2022	No	Yes (Appendix 3)	Yes	Yes
Aug 2023 / Nagoya	Effects of Plasma Treatment Speed on the Mechanical Properties of Flax/Epoxy Composite	International Conference on Environment Pollution and Prevention (ICEPP - 23)	No	Yes (Appendix 4)	Yes	Yes
Aug 2023 / Tokyo	Flame Retardancy of Epoxy-based Composites containing Phosphate Fertilizer	International Conference on Plant & Soil Science (ICPSS - 23)	No	Yes (Appendix 5)	Yes	Yes

10. Whether Research Experience And New Knowledge Has Been Transferred / Has Contributed To Teaching And Learning

(Please elaborate)

The findings of the project especially on the new treatment methods on natural fiber reinforced composites are introduced in the courses of SEHH2249 Engineering Materials, SEHS4618 Aircraft Systems as well as SEHS4611 Final Year Capstone Project.

11. Student(s) Trained

(Please attach a copy of the title page of the thesis)

Name	Degree Registered for	Date of Registration	Date of Thesis Submission / Graduation
	Bachelor of Engineering (Hon) in Mechanical Engineering, PolyU SPEED	1 September 2020	7 May 2021 ^{Notes} (As Final Year Projects)
	Bachelor of Engineering (Hon) in Mechanical Engineering, PolyU SPEED	1 September 2021	13 May 2022 (As Final Year Projects)
	Bachelor of Engineering (Hon) in Mechanical Engineering, PolyU SPEED	1 September 2022	5 May 2023 (As Final Year Projects)

^{Notes} The final year project was commenced in the last semester of final year of their studies, i.e. Jan – May 2021.

12. Other Impact

(e.g. award of patents or prizes, collaboration with other research institutions, technology transfer, teaching enhancement, etc.)

Upon completion of this research project, prototypes of “green” composite materials, their corresponding APP treatment techniques, and the innovative use of micro GO/CG particles for property enhancement have been developed. Based on the acquired knowledge foundation of technical know-how, the PI has liaised with On Fat Lung Innovative Resources Limited for research collaboration on a novel design of flame-retardant playground tiles using recycled waste tires.

13. Statistics on Research Outputs

	Peer-reviewed Journal Publications	Conference Papers	Scholarly Books, Monographs and Chapters	Patents Awarded	Other Research Outputs (please specify)	
No. of outputs arising directly from this research project	2 (Under Review/ Under Preparation)	4	N/A	N/A	Type	No.
					N/A	N/A

14. Public Access Of Completion Report

(Please specify the information, if any, that cannot be provided for public access and give the reasons.)

Information that Cannot Be Provided for Public Access	Reasons
N/A	