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Development of Bio-based Flame Retardants using Recovered Biopolymers from Wastewater Sludge

Nam Kyeun Kim[1], **Krishnan Jayaraman**[1], **Debes Bhattacharyya**[1], **Mark van Loosdrecht**[2] **and Yuemei Lin**[2]

[1]*Centre for Advanced Materials Manufacturing and Design, Department of Mechanical & Mechatronics Engineering, The University of Auckland, New Zealand*
[2]*Department of Biotechnology, Delft University of Technology, the Netherlands*

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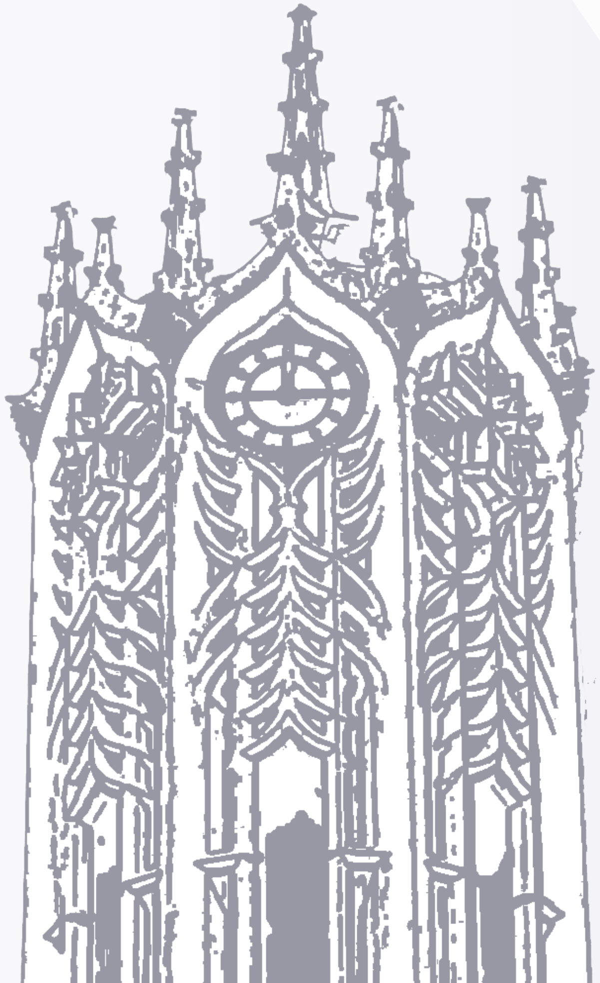
- Experimental
- Results and Discussion
- Conclusions



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Background


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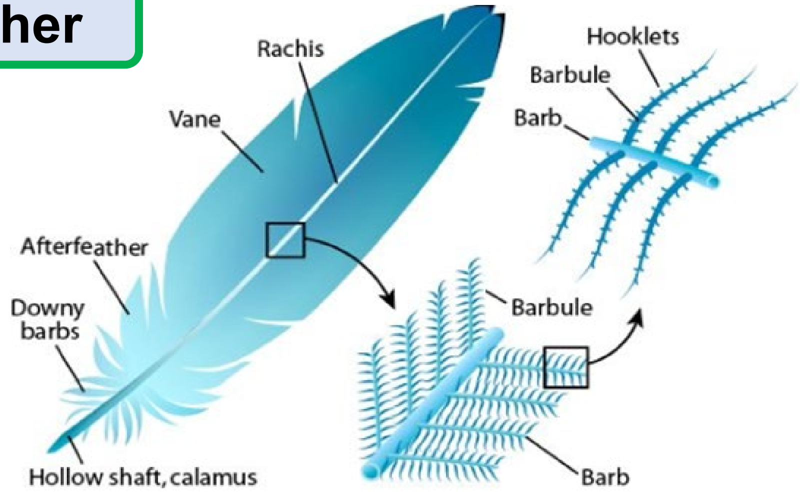
(Fox News-Investigative Committee of the Russian Federation via Storyful)

- **Natural fibre based flame retardants**

Feather



Fighting fires with feathers - chicken waste gets a new use as fire-retardant

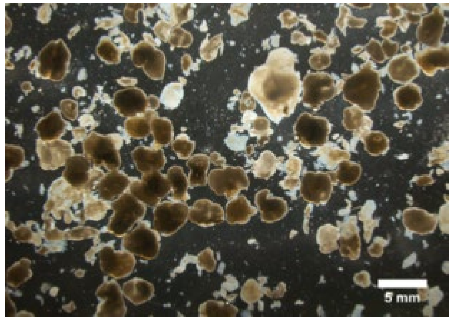
Rebecca Black · 09:38, Jul 26 2019

(D. Jung, D. Bhattacharyya, ACS Sustain. Chem. Eng (2019) 7(23) 19072)

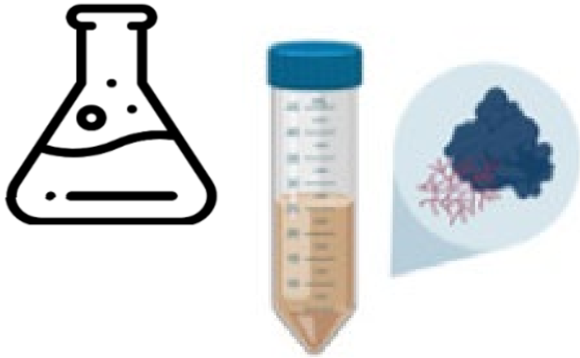
Background

➢ Extracellular Polymeric Substances (EPS)

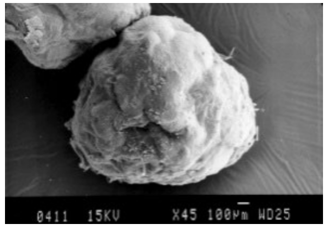
Wastewater treatment plants



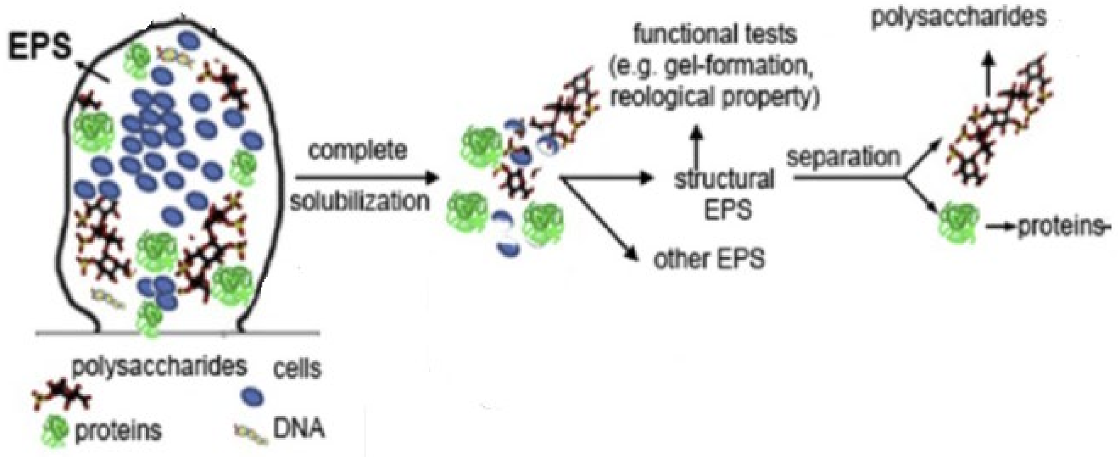
Waste sludge



EPS extraction



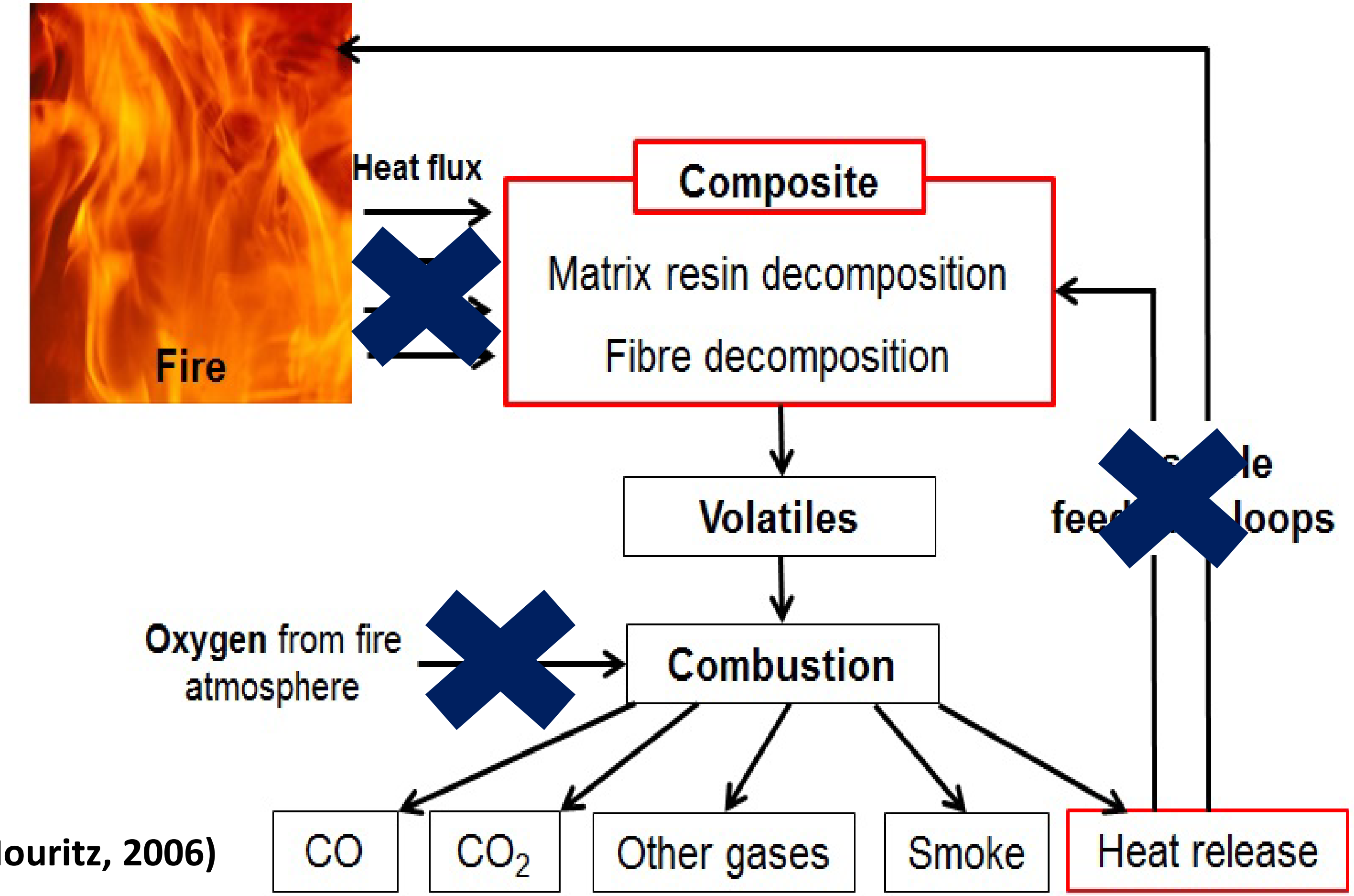
EPS



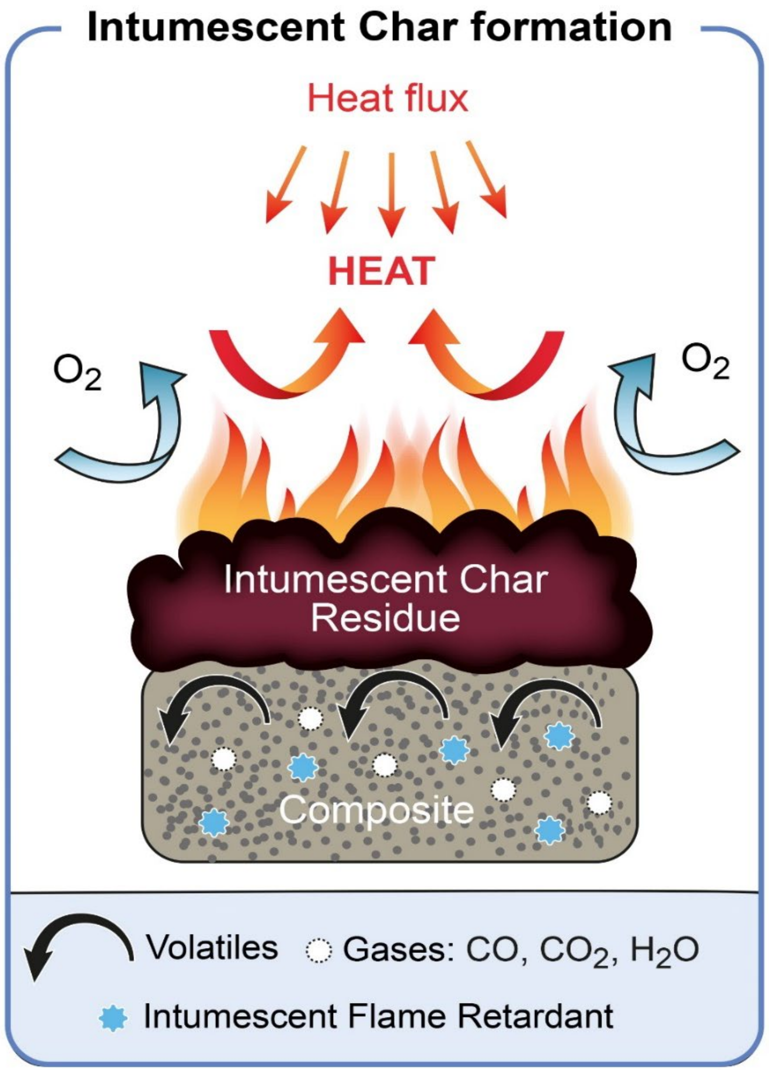
- ➢ **Potential Applications of EPS**
  - ➢ **Paper coating**
  - ➢ **Flame retardant**
  - ➢ **Cement curing**

(*Seviour, Derlon….Loosdrecht, Lin et al. Water Res (2019) 151.*)

Combustion Cycle / Flame Retardation



(Mouritz, 2006)



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EPS Extraction and Phosphorylation

➢ EPS Extraction

Wastewater treatment plant

Wastewater treatment processes → Lime stabilisation

Biosolid

EPS powder

EPS extraction process

Storage at fridge (4°C) → Na_2CO_3 Solution mixing → Stirring in a water bath (80°C) → Centrifugation (4°C) → pH adjust with 1M HCl → Centrifugation (4°C) → Pellet re-dissolving with NaOH at pH 8.5 → Freezing (-50 °C)

EPS extraction process diagram using centrifugal force

➢ Phosphorylation process

EPS solution in distilled water (40 °C)

↓

Adding trisodium trimetaphosphate

↓

Adjust solution to pH12 using 1M NaOH

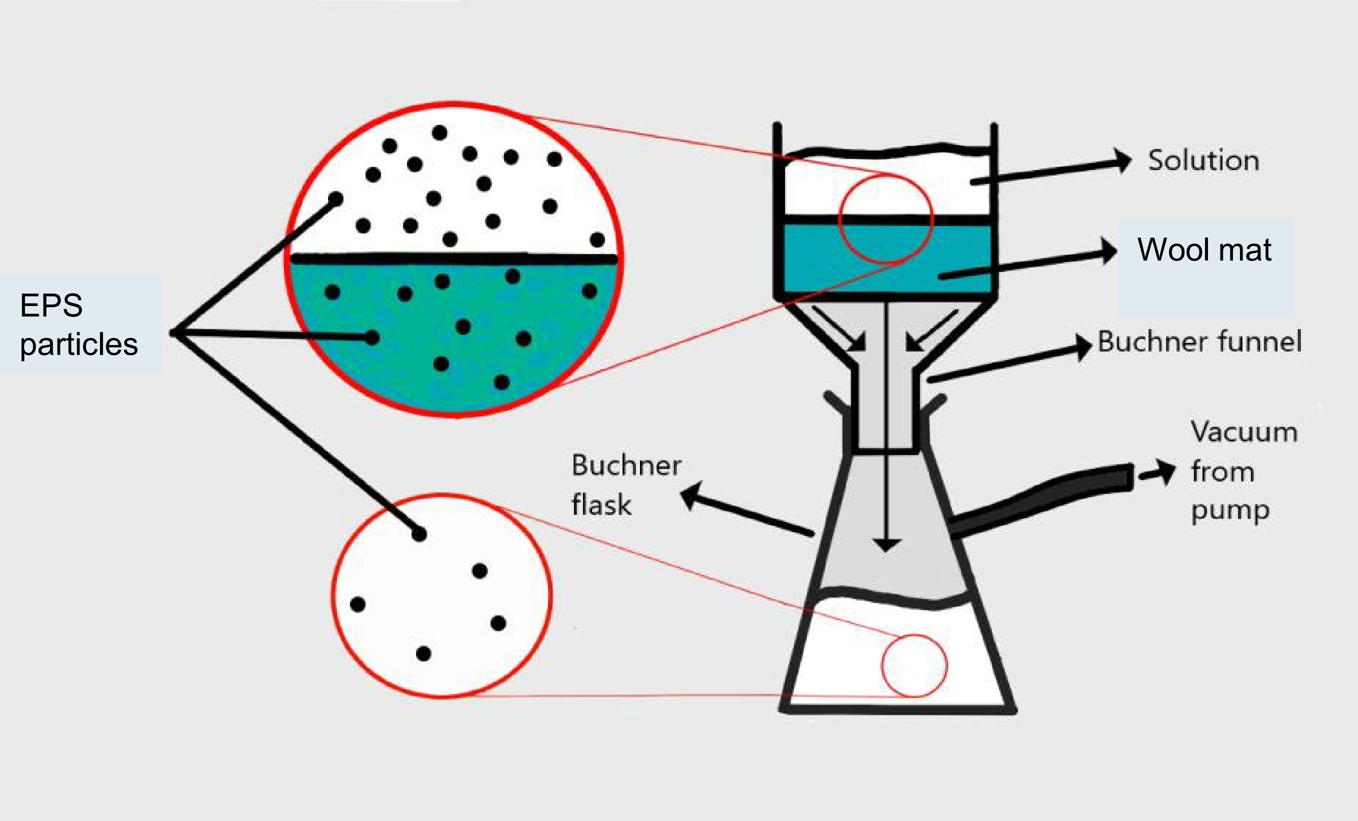
EPS / STMP

EPS coated Wool Felt

➢ Wool felt

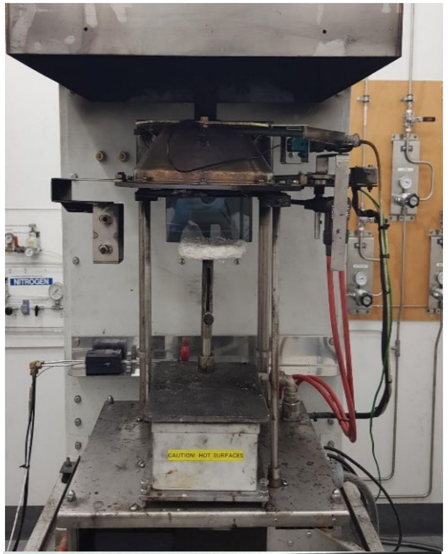
- Medium density (250 kg/m^3) wool felt for filtration, cladding and sealing applications

➢ Vacuum-assisted Dip Coating Method



Characterisation

- **Fourier Transform Infrared Spectroscopy**
- **Thermogravimetric analysis (TGA)**
- **Vertical burn test (ASTM D3801)**
- **Cone calorimeter**
- **Scanning electron microscope (SEM)**



- **ASTM E1354 standard**
- **50 kW/m² heat flux**
- **Fire reaction properties**
  - Time to ignition (TTI)
  - Peak heat release rate (PHRR)
  - Total heat release (THR)
  - Total smoke production (TSP)

CONTENT

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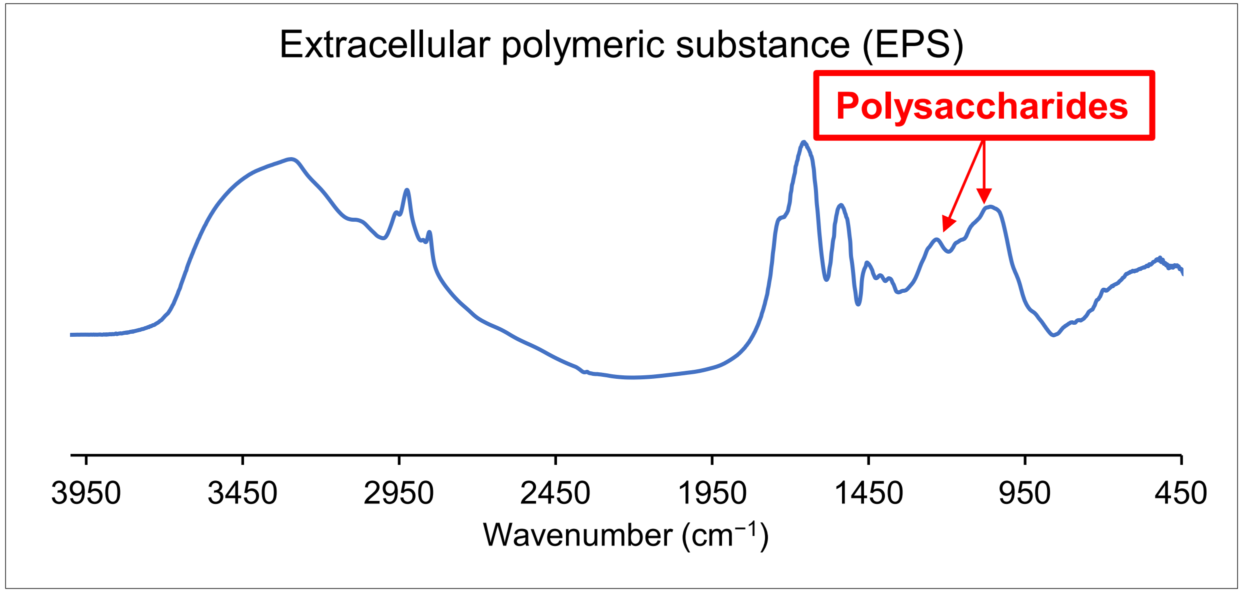
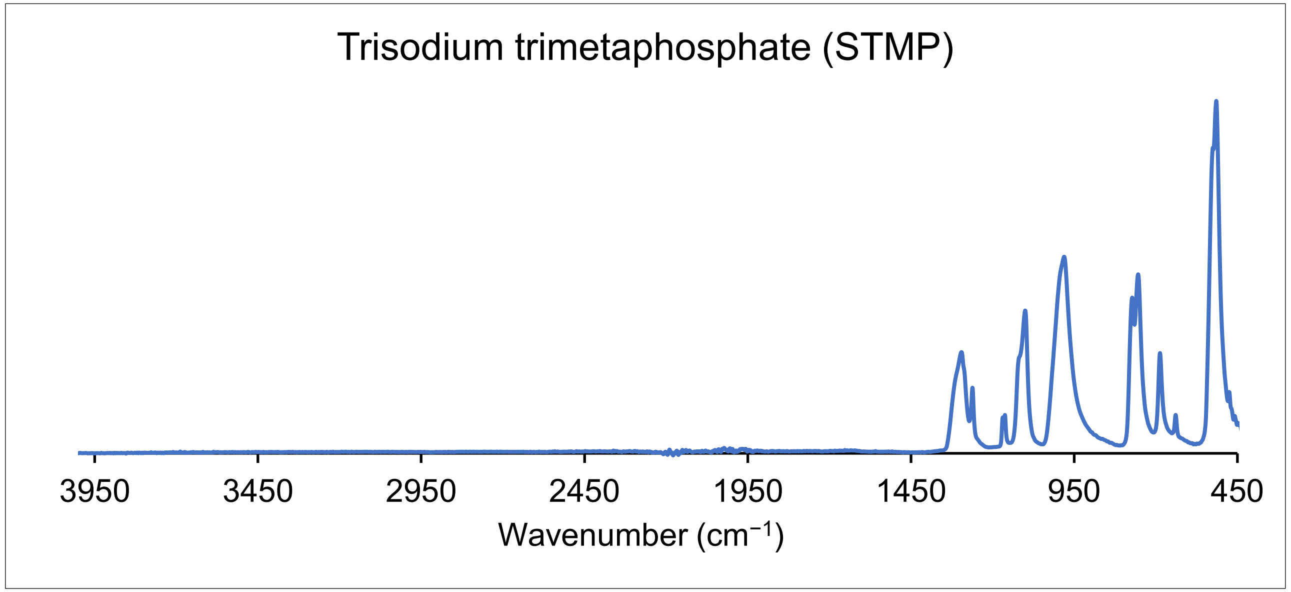
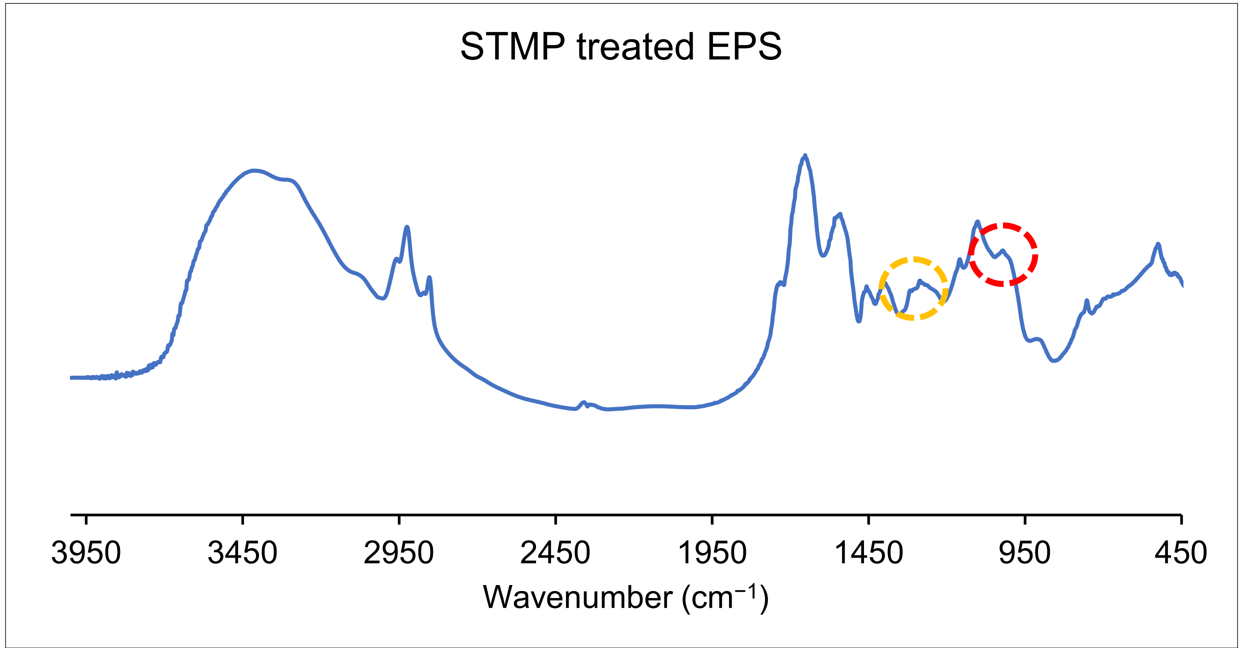
Fourier Transform Infrared Spectroscopy

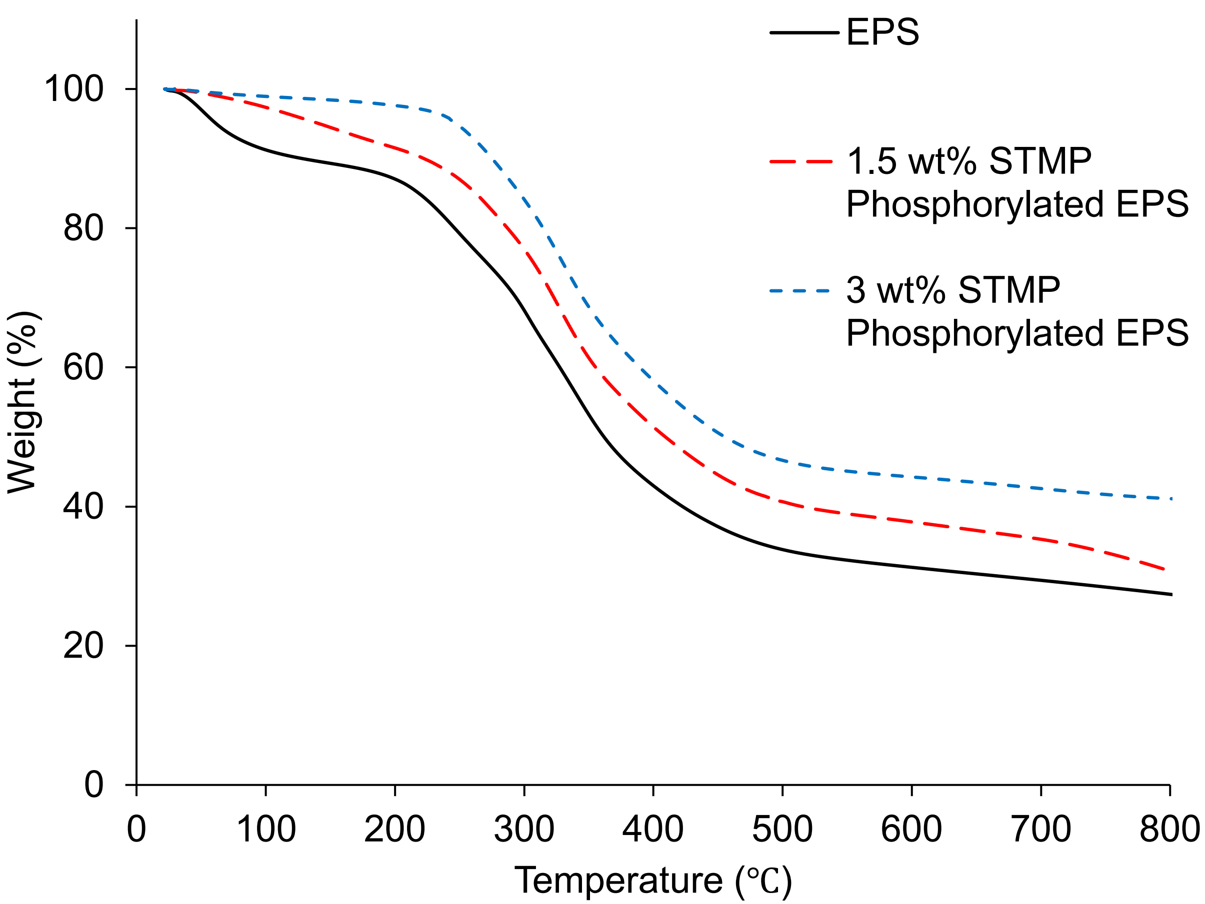






- 1072 cm^{-1} and 1233 cm^{-1} : Polysaccharide
- The new peak at 998.7 cm^{-1} indicates that the P-OH group has successfully bonded with EPS.
- The new peak at 1286.44 cm^{-1} indicates that the EPS has got P=O stretching vibration because of adding STMP.

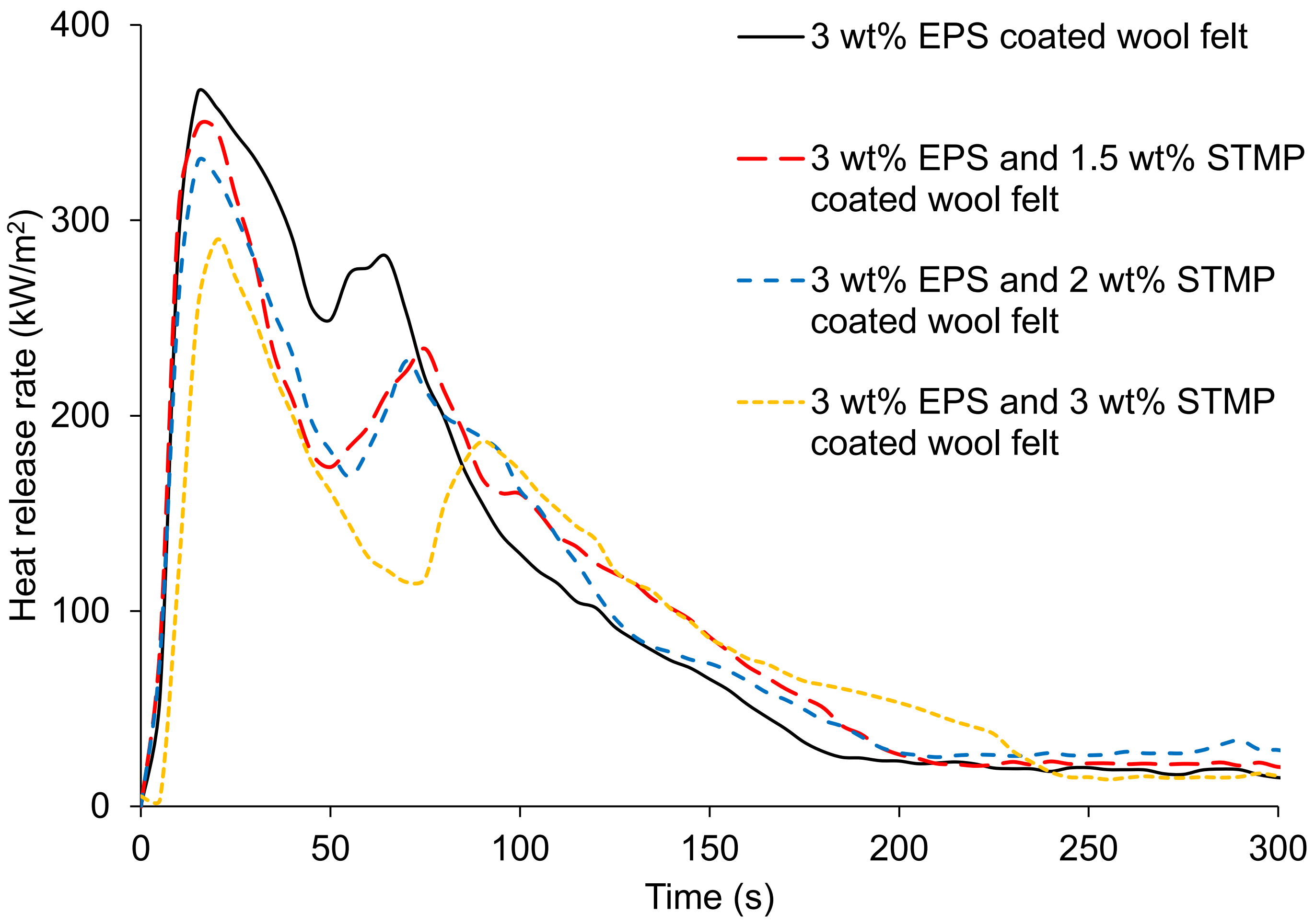
Thermogravimetric Analysis

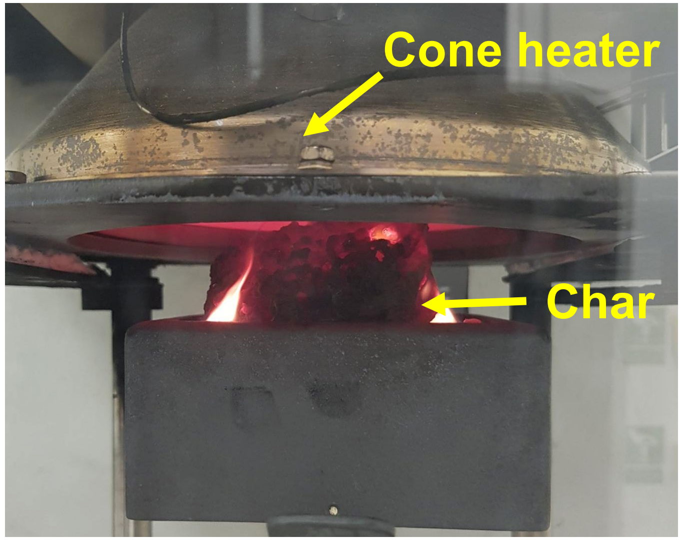


Sample	Amount of residue at 800 °C (wt%)
EPS from wastewater sludge	27.4
1.5 wt% STMP treated EPS	30.7
3 wt% STMP treated EPS	41.1
Wool	24.1
Ammonium polyphosphate	29

Fire Reaction Properties

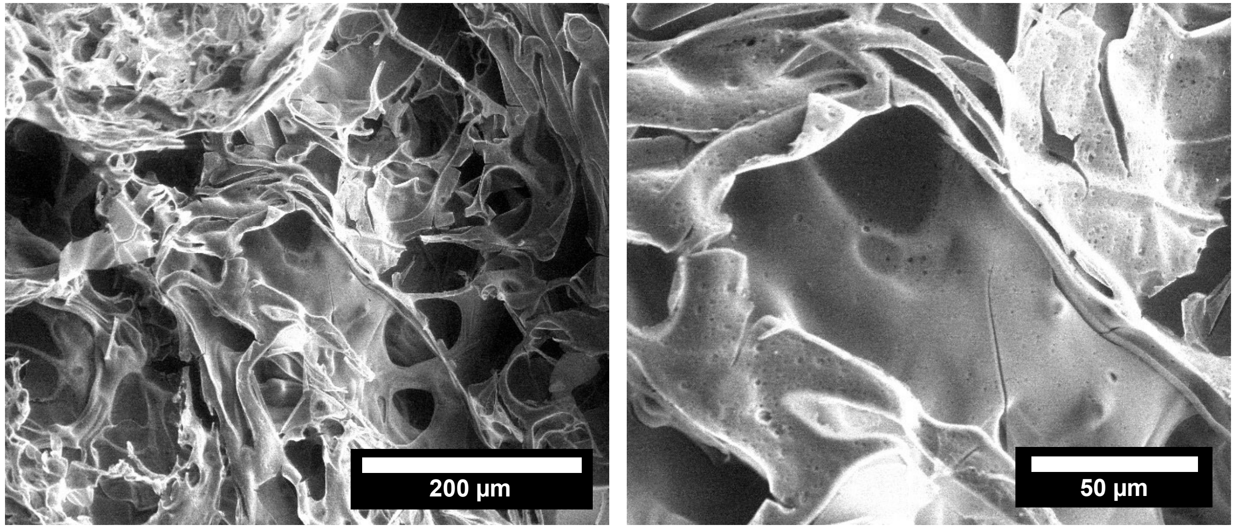
➢ **Heat release rate**



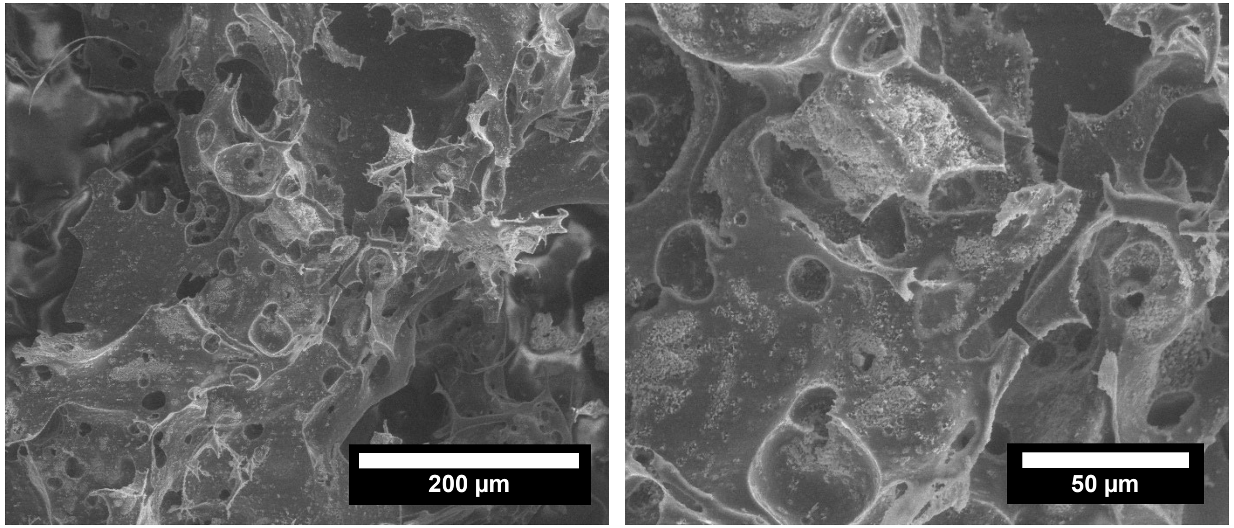


Sample	CO yield (kg/kg)	CO_2 yield (kg/kg)
Wool felt	0.0108	1.21
3 wt% EPS wool felt	0.0151	1.33
3 wt% EPS/1.5 wt% STMP wool felt	0.0155	1.3
3 wt% EPS/2 wt% STMP wool felt	0.0159	1.24
3 wt% EPS/3 wt% STMP wool felt	0.0109	1.08

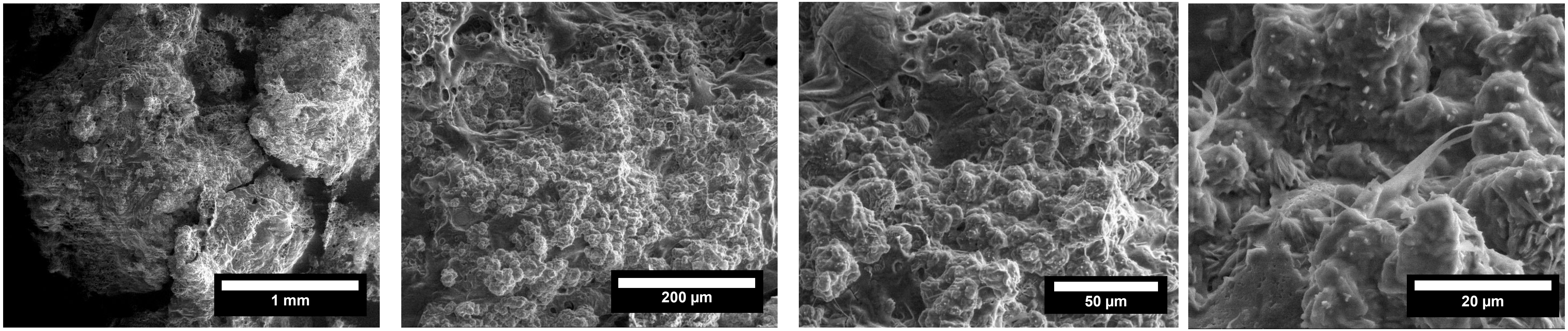
Microstructures of Char



Wool felt



Wool/EPS felt



Wool/EPS/STMP felt

Vertical Burn

Wool felt

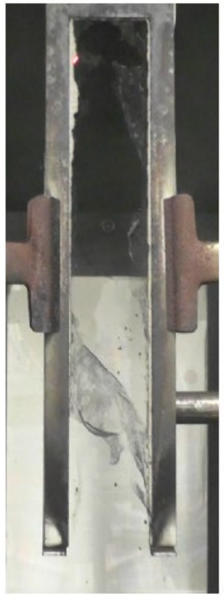
EPS coated Wool felt

EPS/STMP1.5 coated Wool felt

EPS/STMP2 coated Wool felt

EPS/STMP3 coated Wool felt

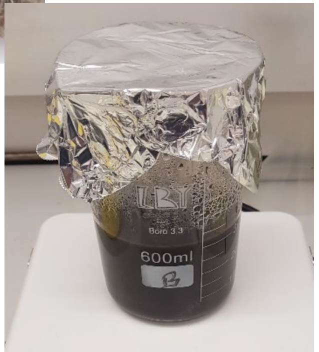
EPS coated Flax Fibre/PP Composites



Untreated Flax

EPS coated Flax

Flax



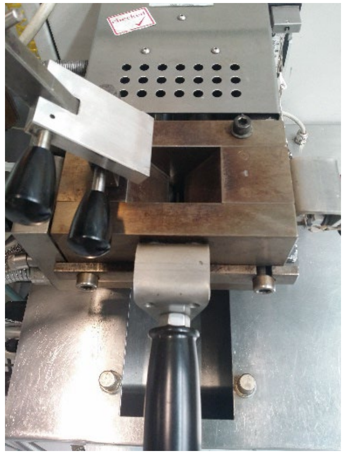
EPS solution

EPS coated flax fibres

~ 5 wt.% EPS absorbed in flax

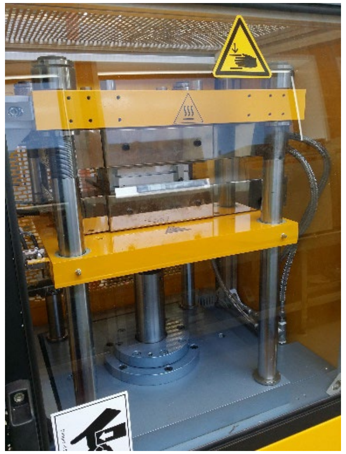
Granulator

Short fibres after grinding



Composite pellets

Melt-blending (compounding)



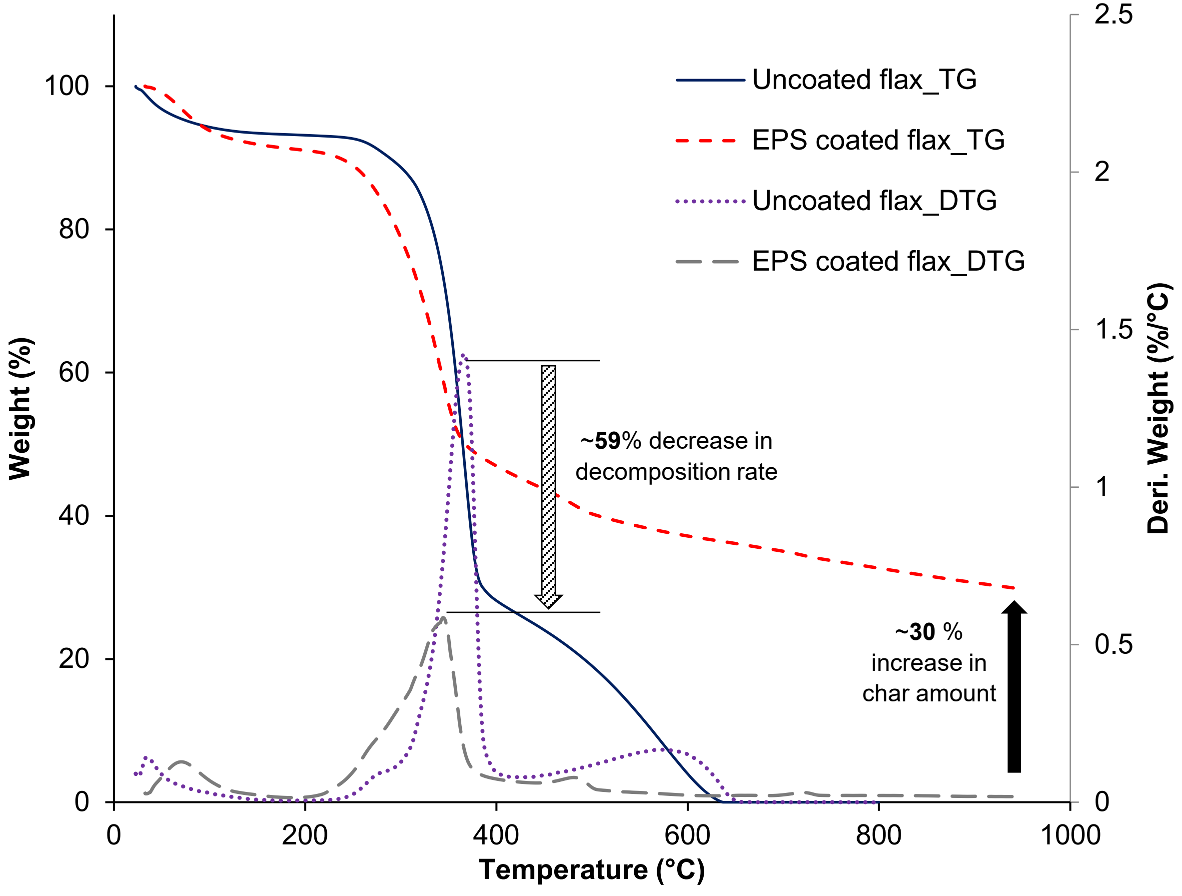
Compression moulding process

EPS coated flax fibre **(30 wt.%)** based composite panels

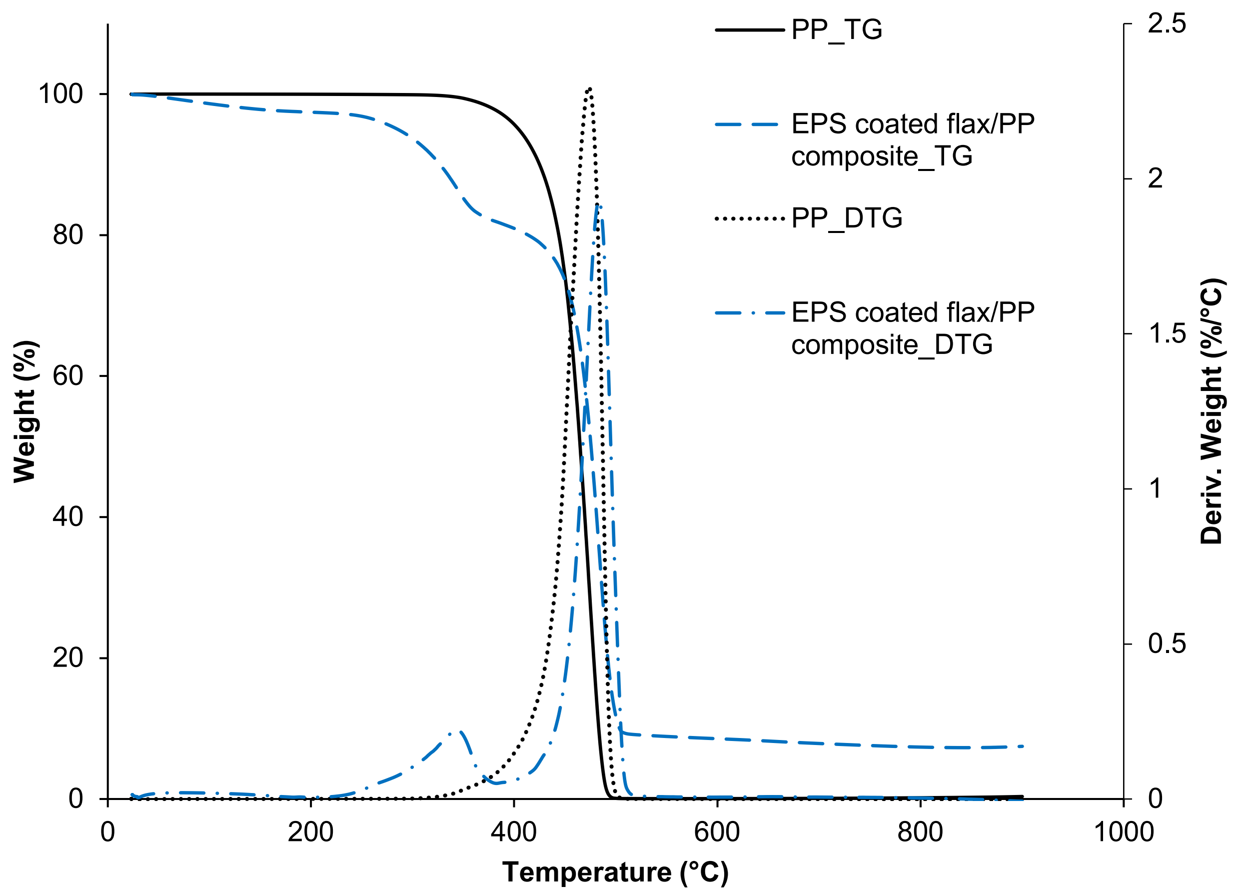
(Kim, Bhattacharyya, Loosdrecht, Lin. Polymer Testing. (2023) Under review)

Thermogravimetric Analysis

➢ EPS coated flax fibres

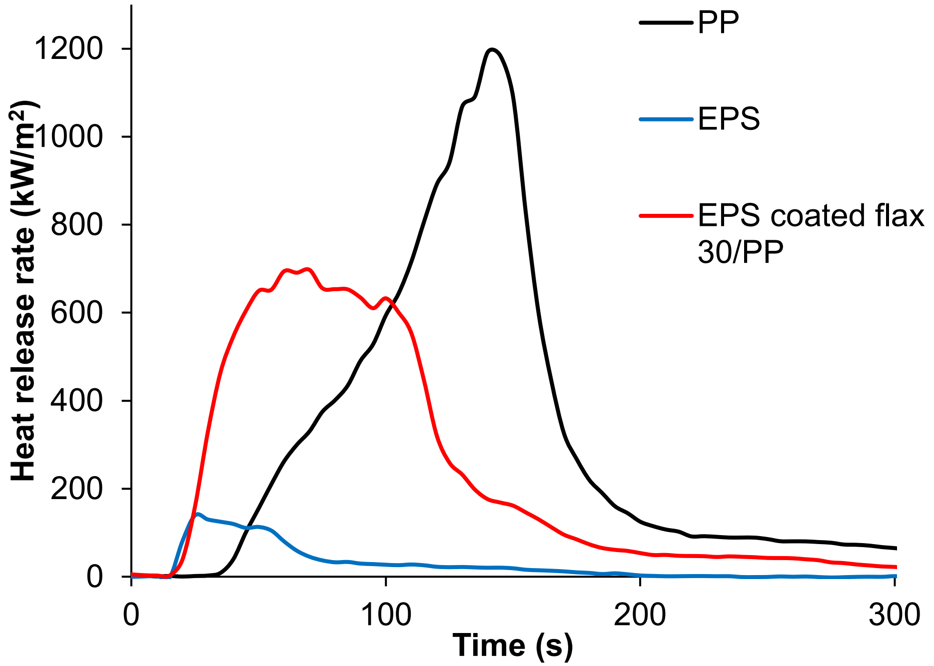


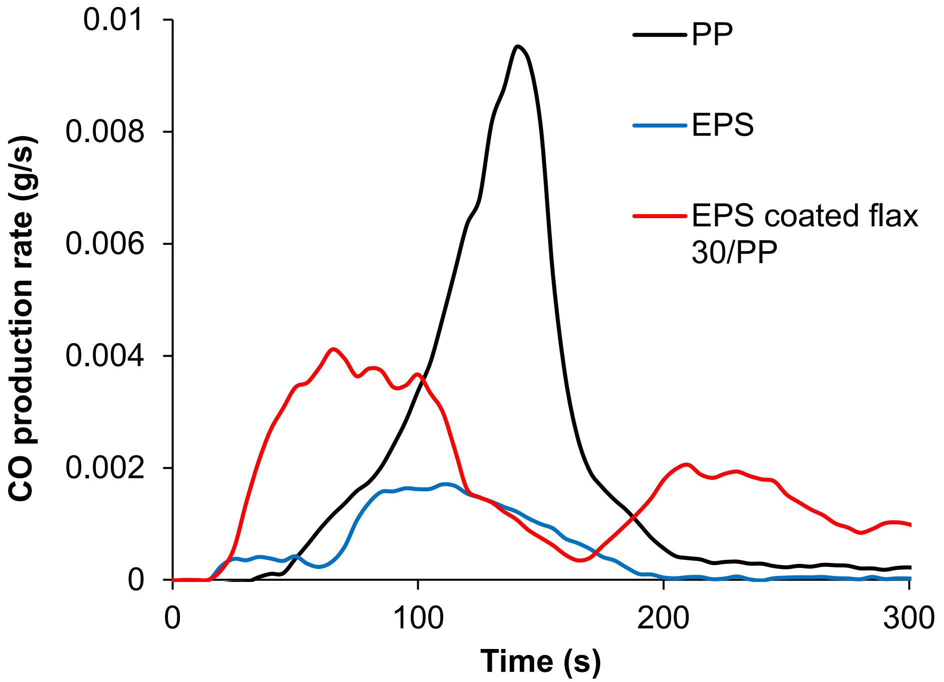
➢ EPS coated flax fibres/PP composite



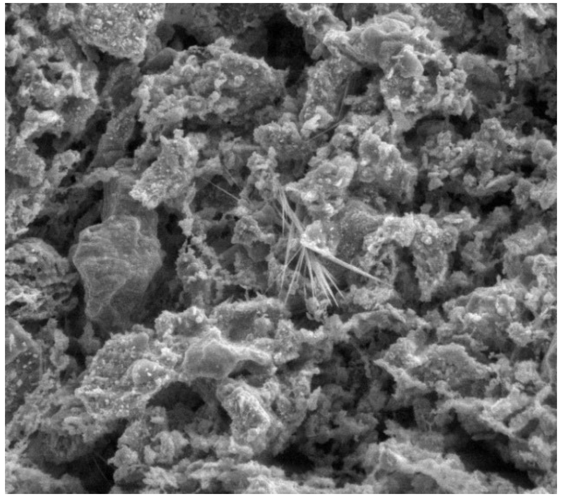
(*Kim, Bhattacharyya, Loosdrecht, Lin. Polymer Testing. (2023) Under review*)

Fire Reaction Properties





	PP	EPS	EPS coated flax/PP
Peak heat release rate (kW/m^2)	1054	163.2	697.3
Total heat release (MJ/m^2)	117	8.8	71.4
CO (kg/kg)	0.029	0.036	0.032
CO_2 (kg/kg)	2.93	1.21	2.28



(Kim, Bhattacharyya, Loosdrecht, Lin. Sci. Total Environ. (2022) 805, 150434 / Kim, Bhattacharyya, Loosdrecht, Lin. Polymer Testing. (2023) Under review)

Conclusions

- The phosphorylation process of EPS created additional phosphorus groups and improved char forming capability of the biopolymer
- The ~ 30 wt% EPS in wool felt achieved the self-extinguishment and reduced peak heat release rate (~18 %) compared to one of wool felt.
- The addition of EPS and STMP into wool felt achieved the remarkable decrease in peak heat release rate (35 %) in comparison with one of wool due to the compact char formation.
- The 30 wt.% EPS coated flax fibre/PP composite achieved the reduction of peak heat release rate (33.8 %) and CO production rate (55.7 %) in comparison with those of PP.
- EPS contributed effective char formation to reduce flammability of wool felt and composite.

THANK YOU

nam.kim@auckland.ac.nz