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The Synergistic Effect of Ionic Liquid-Modified Expandable Graphite and Intumescent Flame-Retardant on Flame-Retardant Rigid Polyurethane Foams

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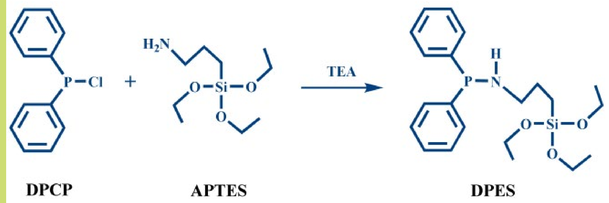
❖Abstract:

Polyurethanes are large molecules formed by combining substances with hydroxyl groups with polyisocyanates through polyaddition reaction for many applications like foams, adhesives, coating, etc. Rigid polyurethane foams (RPUFs) are created for soundproofing and thermal insulation material. In this study, RPUFs have been modified by using nitrogen-phosphorus-containing flame-retardant, an ionic liquid (IL), and expandable graphite. To confirm the successful synthesis Fourier transform infrared (FT-IR) spectra and nuclear magnetic resonance (NMR) spectroscopy have been performed. RPUFs were mixed with IL-EG and DPES to see how they change the structure of the pore and improve the density and strength of the foam's how it breaks down at high temperatures (thermal decomposition). The tests showed that IL-EG/DPES was better in thermal stability at high temperatures and had a great ability to grow and burn. When IL-EG and DPES were mixed in a 1:1 mixture, the flame retardant RPUF had the most flame retardancy, the best flame-retardant performance, and the highest compressive strength also when the ratio of IL-EG/DPES shows the heat release rate (HRR), and smoke release rate (SRR) decreased significantly. The time to ignition (TTI) shows an increasing trend with high wt.% of flame-retardant. This happened because the IL-EG and DPES formed hydrogen bonds with each other, and the new flame-resistant coating on the RPUF surface stopped gas or heat from getting into the PU matrix.

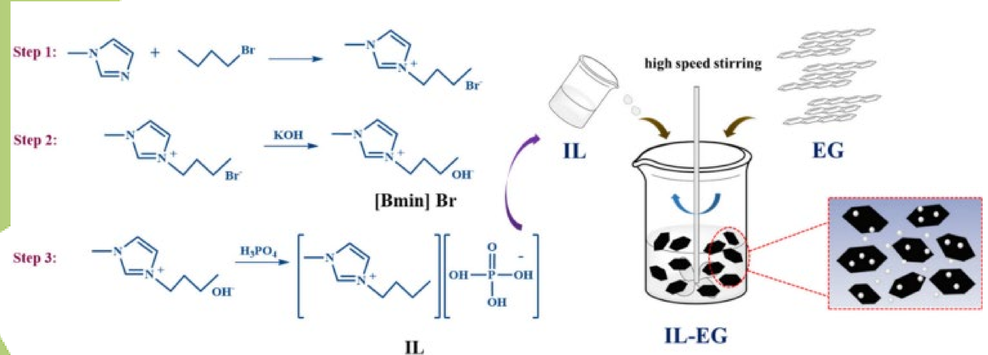
❖ Introduction:

- Rigid polyurethane foams (RPUFs) have been widely applied in thermal insulation, sound insulation shock resistance, etc., on account of their foam structure, low thermal conductivity, excellent specific strength, and low density
- However, RPUFs require flame-retardants due to their inflammability and rapid flame spread. Various flame-retardant additives, such as modified phosphorus, expanded graphite(EG), ammonium polyphosphate, melamine, and intumescent flame-retardants, are used to enhance RPUFs' flame-retardancy. Silicon flame-retardants have gained popularity for their high efficiency, environmental friendliness, anti dripping effects, and smoke suppression.
- This study synthesized a novel halogen-free intumescent flame-retardant, DPES, and combined it with ionic liquid-modified expandable graphite (IL-EG) to improve the mechanical properties of RPUFs.

❖Preparation of DPES



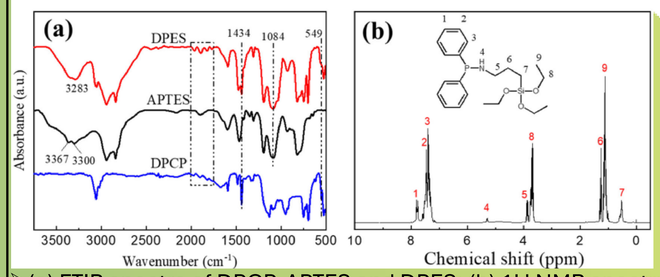
❖Preparation of IL-EG



❖Formulation of RPUFs

Sample	P0 (phpp [a])	P1 (phpp)	P2 (phpp)	P3 (phpp)	P4 (phpp)	P5 (phpp)
HF-4110H	70	70	70	70	70	70
HF-4110	30	30	30	30	30	30
H_2O	3	3	3	3	3	3
AK-8803	2	2	2	2	2	2
A33	2	2	2	2	2	2
GI	1.0	1.0	1.0	1.0	1.0	1.0
PAPI	138	138	138	138	138	138
IL-EG	0	0	5	10	15	20
DPES	0	20	15	10	5	0

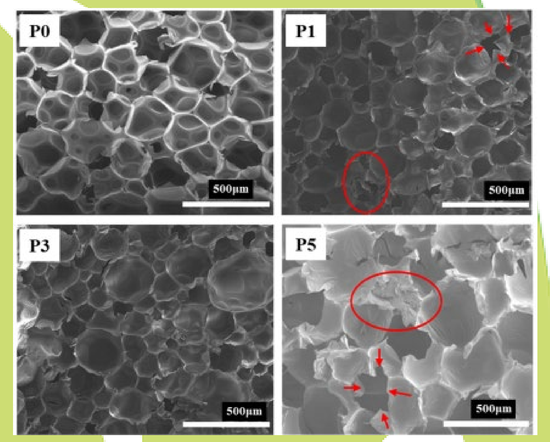
❖Characterization of DPES



➢(a) FTIR spectra of DPCP, APTES and DPES; (b) 1H NMR spectrum of DPES.

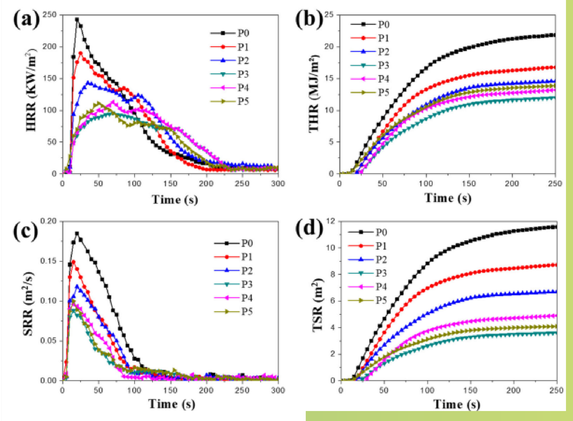
Element	C	H	N	P	Si
Theoretical value (wt %)	61.37	7.97	4.03	7.67	6.93
Measured value (wt %)	61.03	7.72	3.64	7.50	7.05

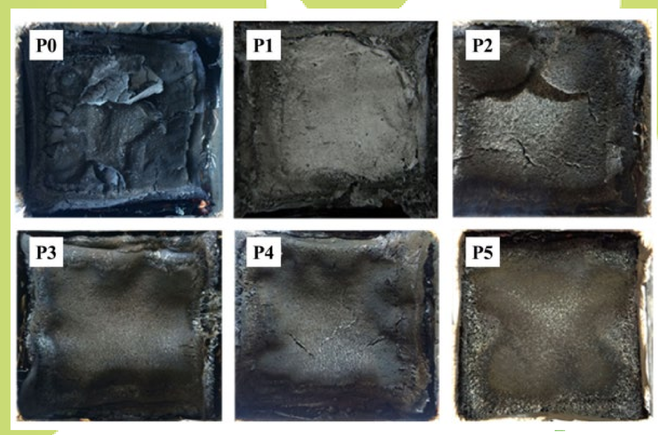
➢Theoretical and measured values of the content of each element in DPES



➢SEM morphology of RPUF with different flame-retardants.

❖Results and discussion





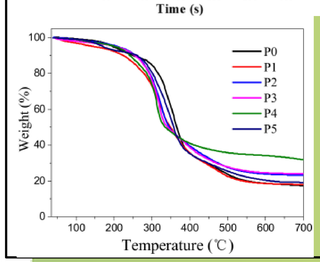
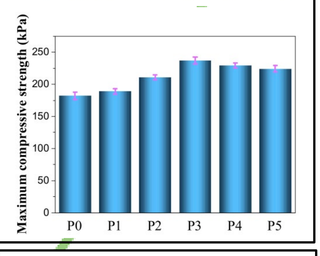


Table 3. TG and DTG data for the various samples.

Sample	$T_{5\%}$ (°C)	$T_{10\%}$ (°C)	$T_{50\%}$ (°C)	T_{onset} (°C)	T_{max} (°C)	Residue at 700 °C (wt%)
P0	196.5	249.8	364.8	180.0	356.5	17.3
P1	145.9	223.4	348.4	125.7	315.1	17.9
P2	210.7	256.7	345.7	196.7	321.2	22.2
P3	205.2	255.2	340.2	188.4	320.7	24.1
P4	203.6	242.3	333.5	189.4	314.4	31.9
P5	171.1	259.8	357.3	150.0	352.0	19.3



❖Conclusion

- Combining flame-retardant IL-EF/DPES optimizes RPUF's structure and flame retardancy, with a 1:1 ratio yielding optimal compressive strength. The modified RPUF containing IL-EG and DPES exhibits reduced PHRR, THR, SRR, and TSR, while enhancing LOI and TTI with a formed char layer, improving flame retardancy.

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