SYNERGISTS FOR HEXAPHENOXYCYCLOTRIPHOSPHAZENE IN CAST PA6

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Introduction

Carbon fiber reinforced (CFR) anionic polyamide 6 (APA6) is a light and at the same time very strong material. Therefore CFR APA6 components are of interest as lightweight construction materials e.g. for aircraft manufacturing. Like many other organic polymers APA6 is flammable and thus it is mandatory to protect the polymer matrix by adding flame retardants (FRs). However, an suitable process for production of CFR APA6 materials such as thermoplastic resin transfer molding (T-RTM) process is highly sensitive to presence of polymer additives such as FRs. E.g. commonly used FRs interrupt the anionic ring opening reaction of ε-caprolactam to form PA6 or are filtered out by the fiber fleece. With hexaphenoxycyclotriphosphazene (HPCTP) we discovered an incorporable FR for CFR APA6.

Results

FRs TESTED IN LAB SCALE

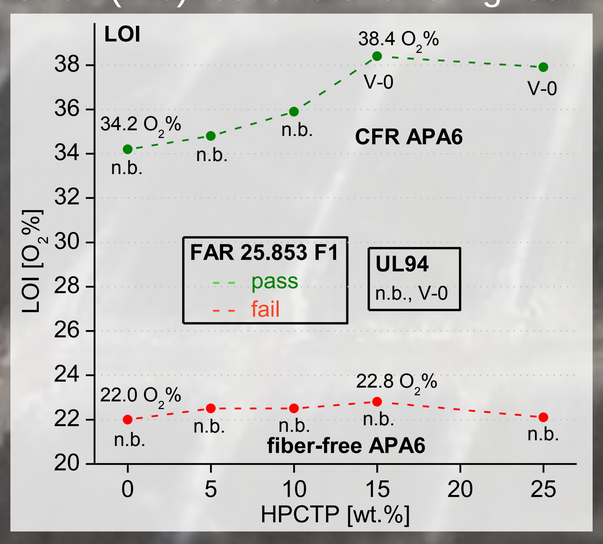
FR requirements for CFR APA6 produced *via* T-RTM:

- **Solubility** in molten ε-Caprolactam
- **No interruption** of the anionic polymerization process

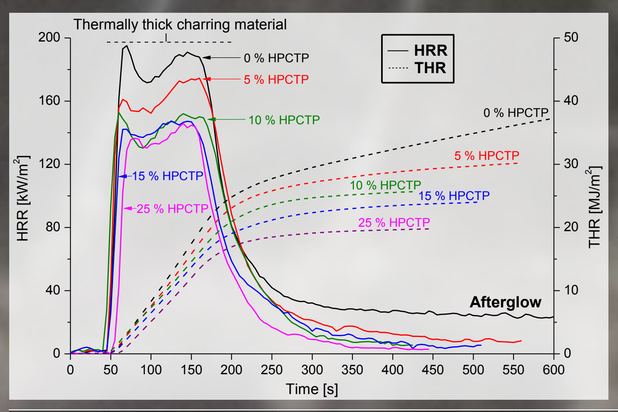
FR	Solubility	No interruption
Rabitle FP110 (HPCTP)	Yes (tested till 50 wt.%)	Yes
Methyl-DOPO	Yes (till 10 wt.%)	Yes (till 5 wt.%)
Aflammit PCO 960	Yes (till 10 wt.%)	Yes (till 1 wt.%)
Exolit AP 462	No	Yes
Apyral 40 CD	No	Yes
Apyral 40 VS1	No	Yes
Magnifin H5 IV	No	Yes
Red phosphorus	No	Yes
Melamine	No	No
Expandable graphit	No	No
Exolit OP 935	No	No
Exolit OP 1312	No	No

FR-TESTS

CFR APA6 with 15 wt.% HPCTP pass FAR and UL94 (V-0) test and shows highest LOI value.



CONE CALORIMETRY OF CFR APA6

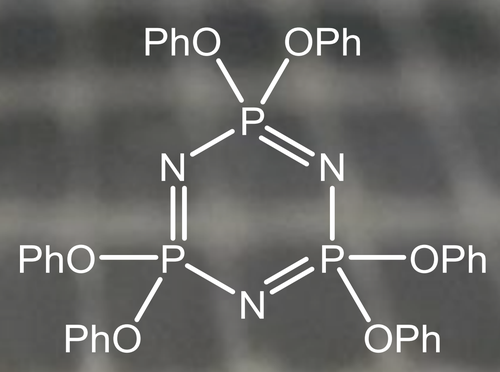


The Heat Release Rate (HRR) decreases with increasing HPCTP load. Without HPCTP, CFR APA6 shows afterglow. Incorporation of HPCTP eliminates afterglow and promotes char formation.

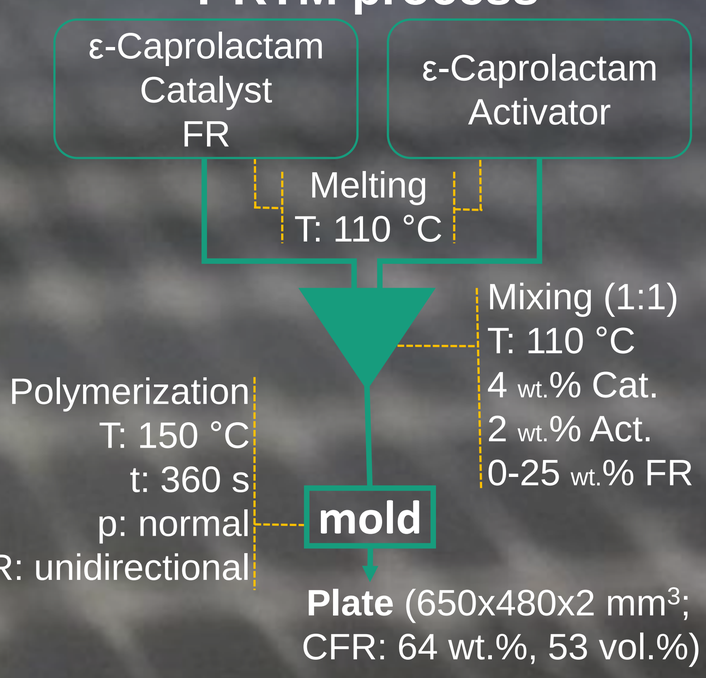
HPCTP [wt.%]	Surface [mm²]	t_{ig} [s]	Peak HRR [kW/m²]	THE [MJ/m²]	TSR [m²/m²]	t_b [s]	MARHE [kW/m²s]	Peak CO [ppm]	Peak CO_2 [%]	Mass loss [wt.%]
0	100x100	47	198	35,6	94	535	126,2	92	0,369	39,8
5	100x100	44	176	29,3	404	450	116,2	201	0,335	30,7
10	100x100	40	155	25,3	528	352	105,6	309	0,302	29,4
15	100x100	47	148	23,4	526	409	97,5	380	0,279	29,5
25	100x100	54	150	19,4	654	380	86,9	564	0,289	28,5

Preparation

Only the HPCTP-FR Rabitle FP110 (Nordmann, Russmann) is applicable in T-RTM process for CFR APA6 with higher loading (see tested FRs).

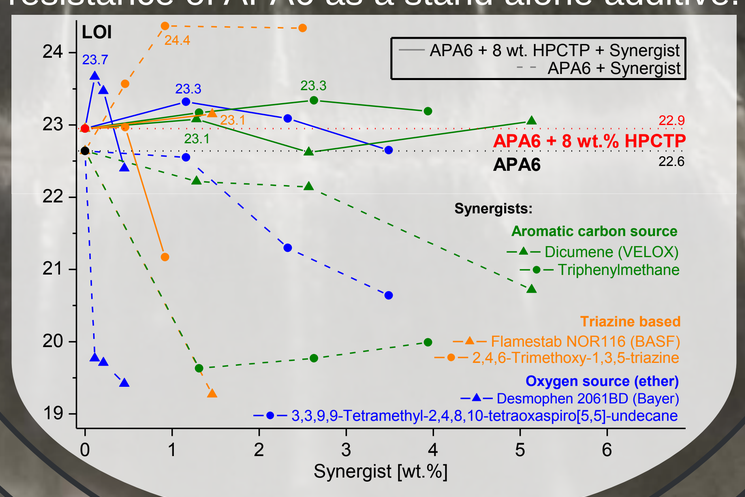


T-RTM process



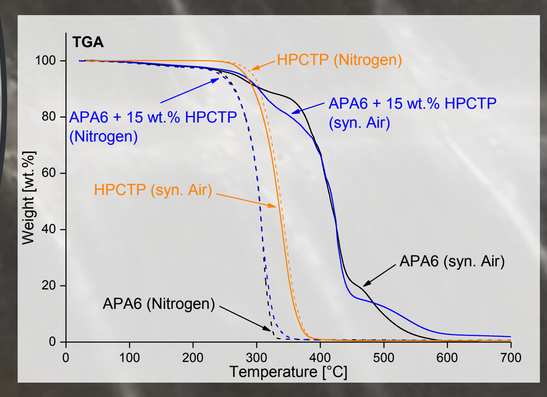
Synergism

For future researches the influence of six additional additives in lab scale experiments was studied. Five of the examined compounds exhibited distinct synergistic effects with HPCTP in APA6 matrix. Although Trimethoxytriazine showed no synergy, it significantly improved flame resistance of APA6 as a stand alone additive.



TGA OF FIBER-FREE APA6

HPCTP promotes in oxygen rich atmosphere (syn. air) the formation of thermally stable char. In an innert atmosphere (Nitrogen) no char formation is observed.



Conclusion

According to TGA and cone calorimetry, char formation is the essential FR mechanism of HPCTP in CFR APA6. Incorporation of only 15 wt.% HPCTP prevents afterglow (cone calorimetry) and allows the material to pass FAR and UL94 (V-0) tests. Following mechnaical properties were obtained for CFR APA5 with 15 wt.% HPCTP (0° orientation).
Crystallinity: 40 %, Impact resistant: 96 kJ/m²
E-mod.: 118 GPa (flex.) 103 GPa (compr.) 134 GPa (Young)
Strength: 1228 MPa (flex.) 463 MPa (compr.) 1471 MPa (tens.)

Reference: C.-C. Höhne, *Master Thesis*, TU Bergakademie Freiberg, **2014**.

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Background: Photo of a CFR APA6 plate *via* T-RTM.

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