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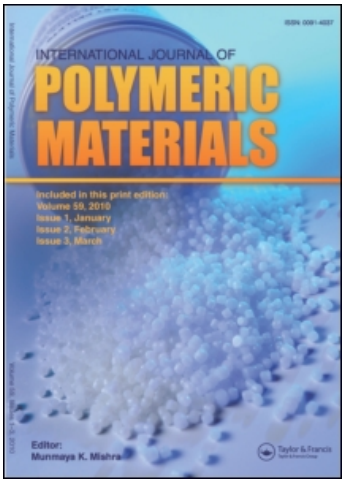

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Polypropylene Flame Retardant System Based on Si-$SnCl_2$

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The effect of addition of various silicon-inorganic compositions on flame retardancy of polypropylene is explored.

KEY WORDS Polypropylene, silicon-inorganic additives, Si-$SnCl_2$, flame retardancy.

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INTRODUCTION

The subject of ecological safeness of polymer flame retardants has become a major problem in modern polymer industry. The different types of polymer flame retardants based on halogens (Cl, Br), heavy and transition metals (Zn, V, Pb, Sb), phosphorus-organic compound may cause an elimination of hazard products during polymer combustion and pyrolysis.

The fire retardancy of polymers can be achieved by different ways: 1) Modifying

TABLE I

Cone data PP-silica systems (heat flux = 20.0 kW/m²)

Pol. System	Initial wt., g	Carbon rel.,	Ignition t., s	Peak RHR, kW/m²	Total heat res.,MJ/m²
PP: Si/$Na_2B_4O_7$ 85:(10/5) (a)	42.0	12.4	131	428.9	341.33
PP: Si/$Na_2B_4O_7$/$SnCl_2$ 85:(8/5/2) (b)	41.5	28.7	394	432.3	194.36
Polypropylene-iso Sp^2	21.7	0.3	217	1266.7	200.84

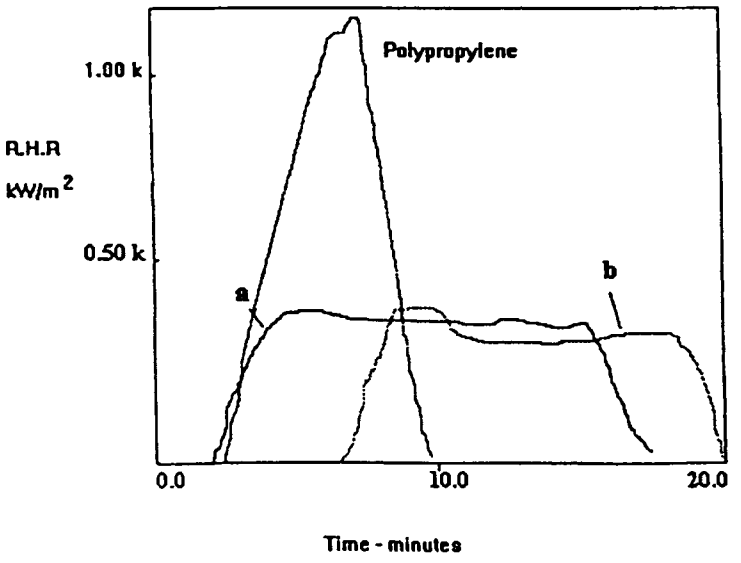

FIGURE 1 Rate of heat release vs. time for polypropylene; polypropylene with $Si/Na_2B_4O_7$ (85%: 10%:5% wt.)—(a); polypropylene with $Si/Na_2B_4O_7/SnCl_2$ (85%:8%:5%:2% wt.)—(b) at heat flux 20 kW/m^2.

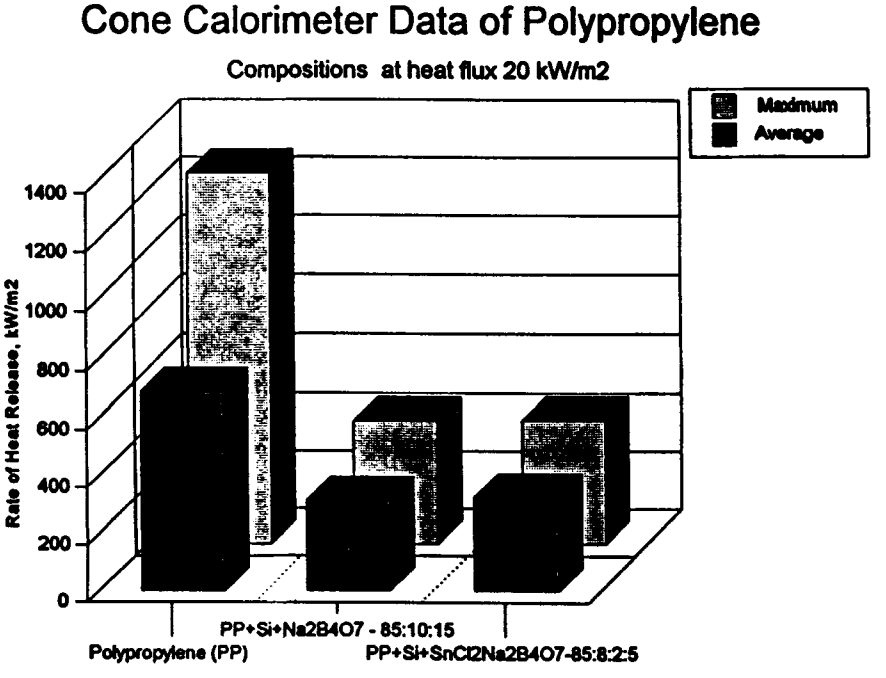

FIGURE 2 Cone calorimeter data of polypropylene-Si compositions at heat flux of 20 kW/m^2 rate of heat release.

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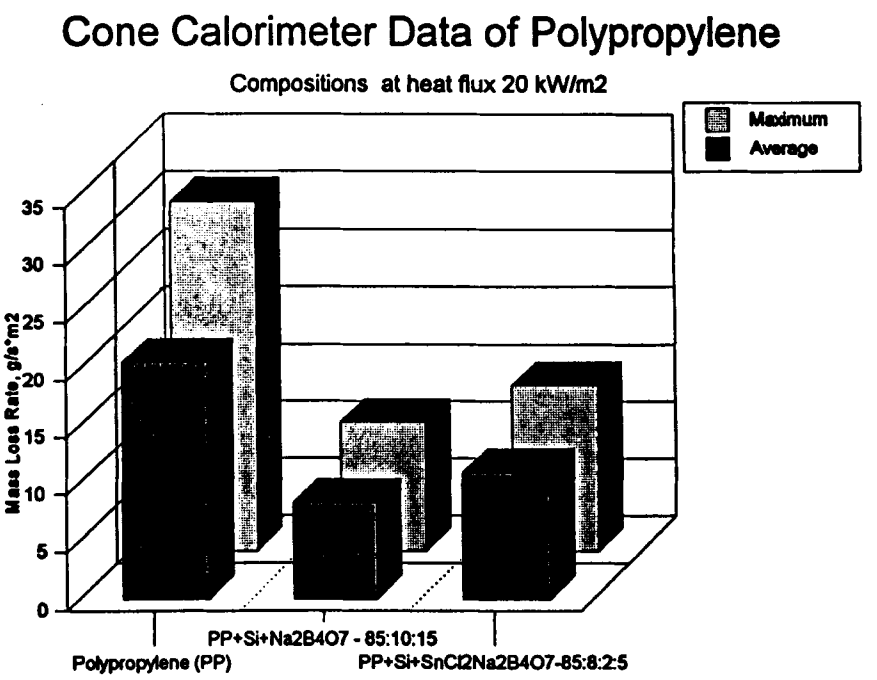

FIGURE 3 Cone calorimeter data of polypropylene-Si compositions at heat flux of 20 kW/m^2 mass loss rate.

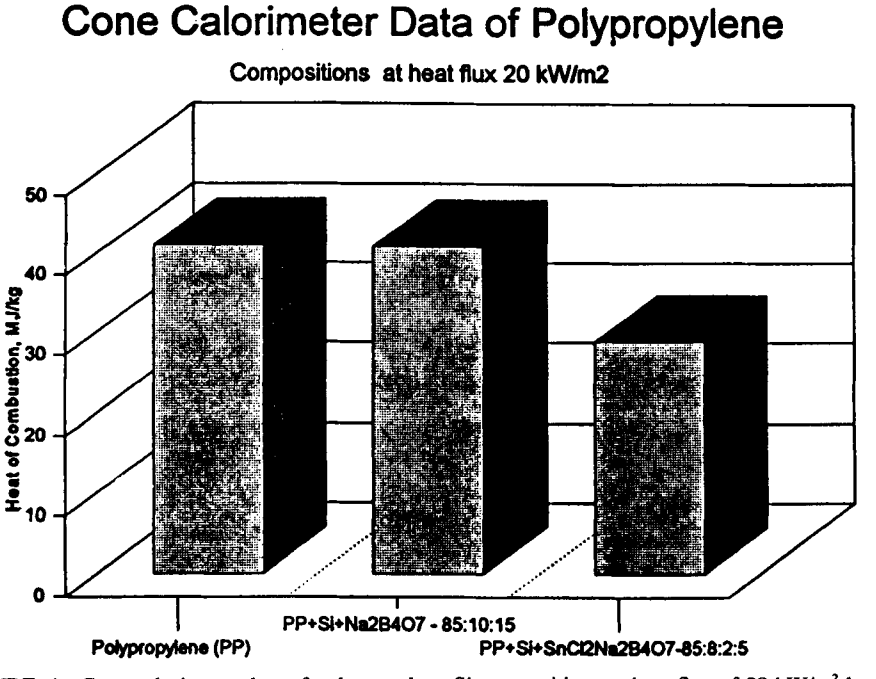

FIGURE 4 Cone calorimeter data of polypropylene-Si compositions at heat flux of 20 kW/m^2 heat of combustion.

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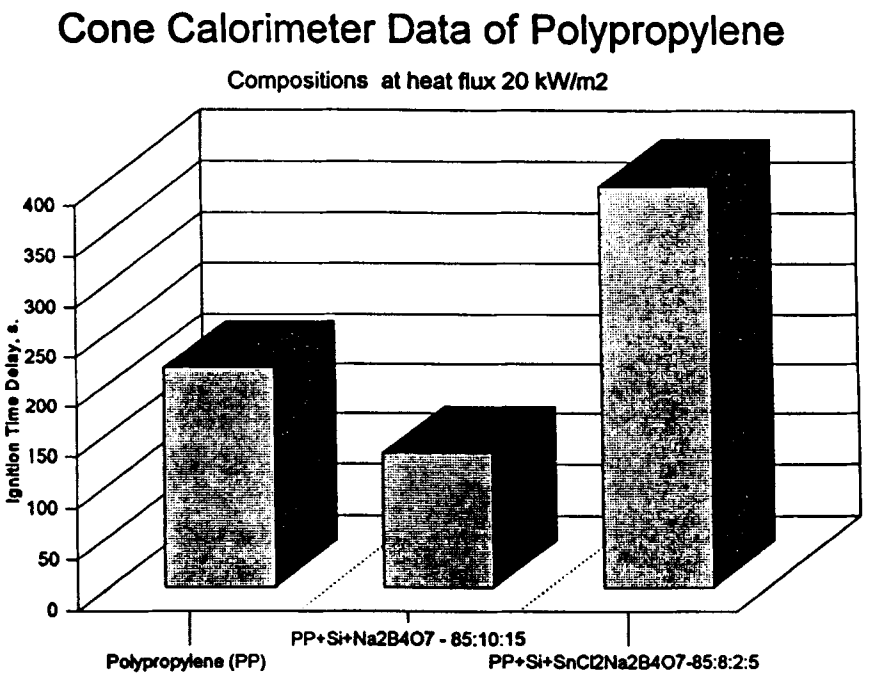

FIGURE 5 Cone calorimeter data of polypropylene-Si compositions at heat flux of 20 kW/m^2 ignition time.

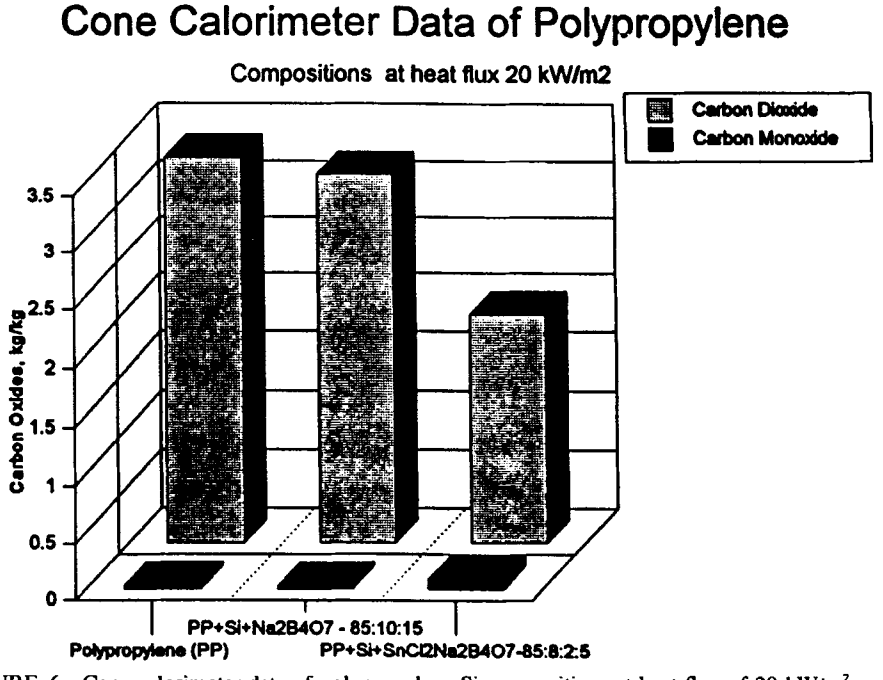

FIGURE 6 Cone calorimeter data of polypropylene-Si compositions at heat flux of 20 kW/m^2 carbon oxides.

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TABLE II

Cone data of Si-PP system at heat flux of 35 kW/m^2

CONE DATA	POLYPROPYLENE	PP+Si+$SnCl_2$ (95:3:2)
Char yield, % wt.	0.0	10.1
Ignition time, sec.	62	91
Peak RHR, kW/m2	1378.0	860.1
Total Heat Release, MJ/m2	332.0	193.7

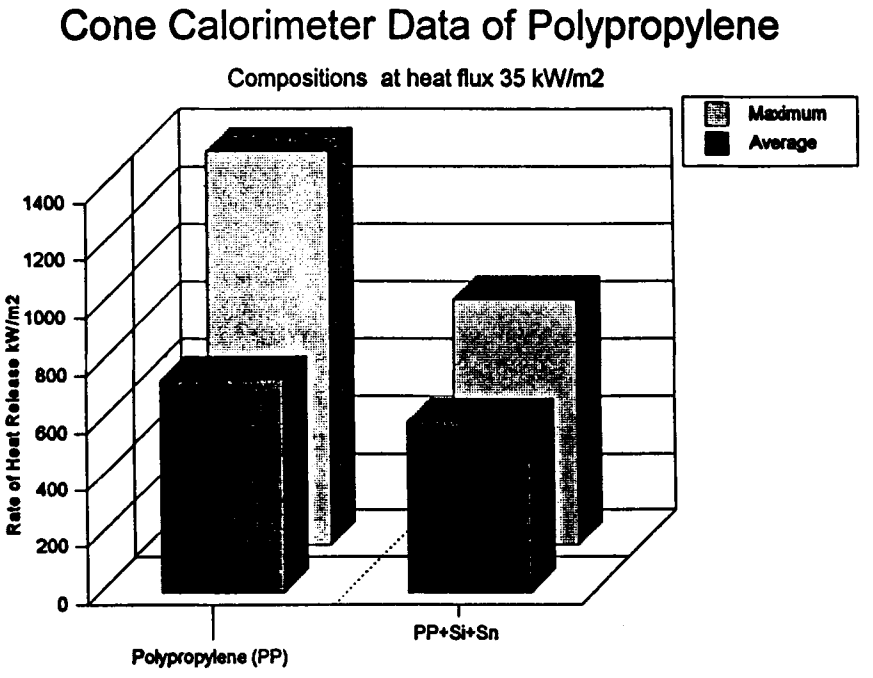

FIGURE 7 Cone calorimeter data of polypropylene-Si composition at heat flux of 35 kW/m^2 rate of heat release.

the pyrolysis scheme: to produce non volatile, or non combustible products that dilute the flame oxygen supply; 2) Smothering the combustion through dilution of the combustible gases, or the occurrence of the barrier (char) which hinders the supply of oxygen; 3) Trapping the active radicals in vapor phase (and eventually in condensed phase); 4) Reducing the thermal conductibility of the material to limit the heat transfer (char).

In our research we have focused on the ways 2, 3 and 4. We propose that silicon-inorganic flame retardant compositions act as gaseous/solid phase inhibitors of polymer combustion.

The proposed flame retardant composition was based on the assumption of trapping the active radicals in vapor phase and eventually in condensed phase. The system we've proposed was silicon-inorganic composition (SI). This is one of the

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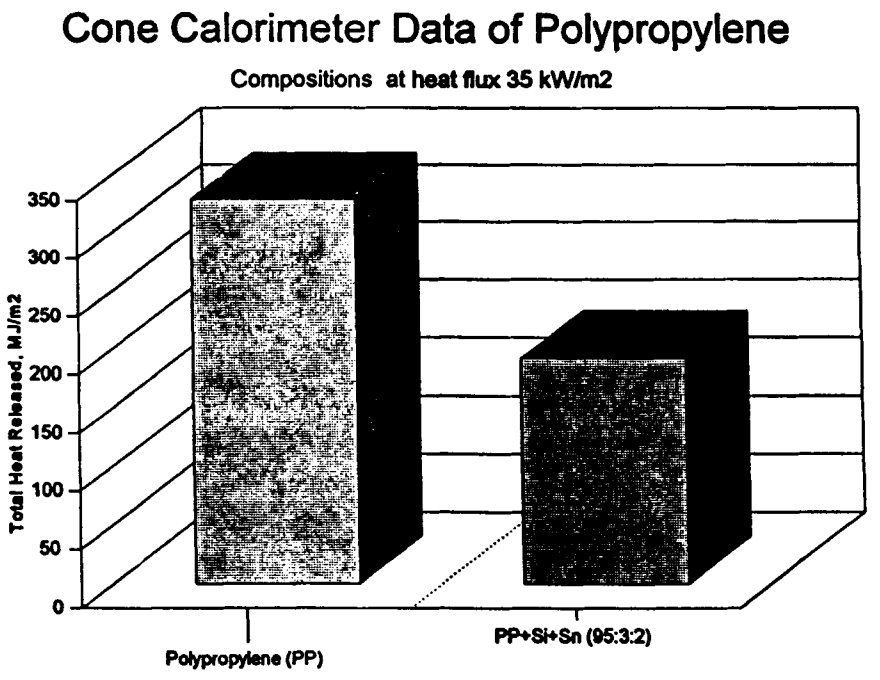

FIGURE 8 Cone calorimeter data of polypropylene-Si composition at heat flux of 35 kW/m^2 total heat release.

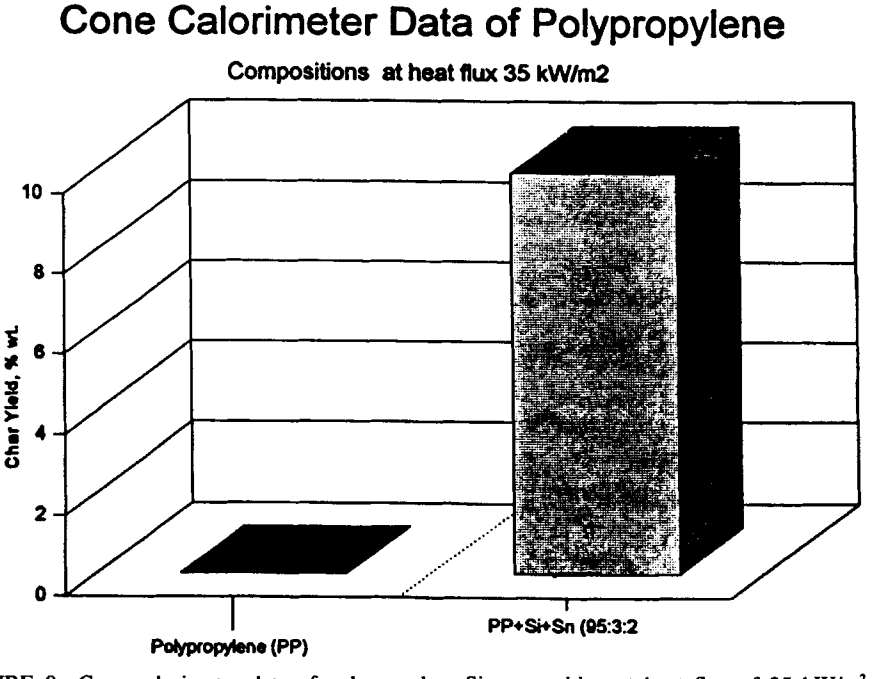

FIGURE 9 Cone calorimeter data of polypropylene-Si composition at heat flux of 35 kW/m^2 char yield.

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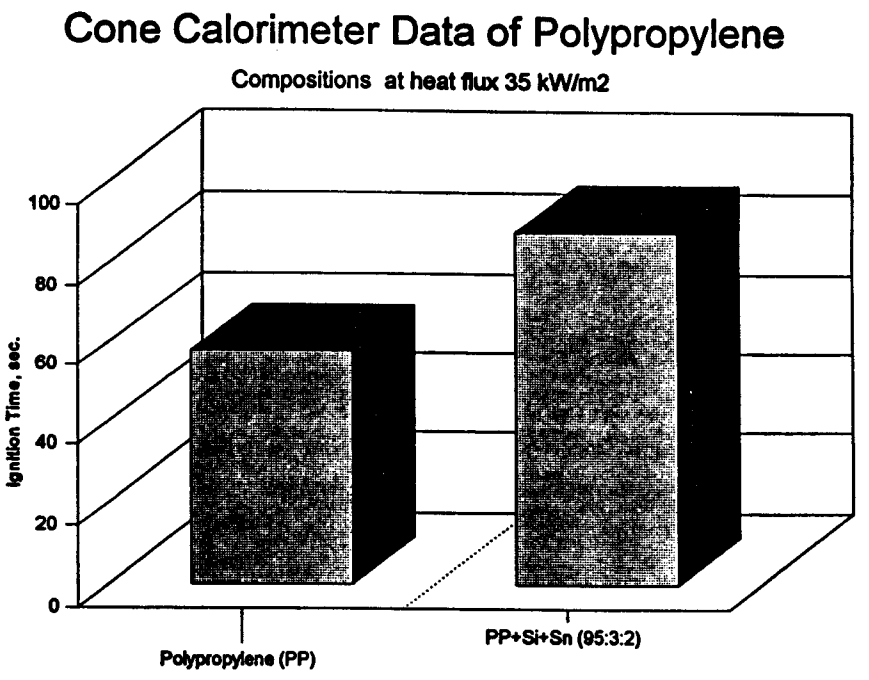

FIGURE 10 Cone calorimeter data of polypropylene-Si composition at heat flux of 35 kW/m^2 ignition time.

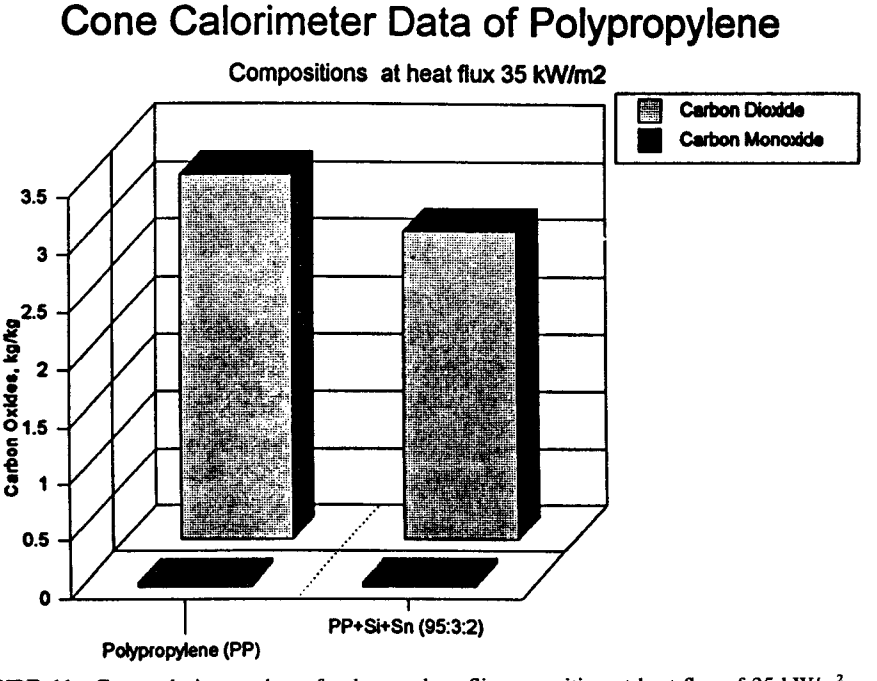

FIGURE 11 Cone calorimeter data of polypropylene-Si composition at heat flux of 35 kW/m^2 carbon oxides.

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most interesting modern flame retardant systems. The possibility of reaction of gaseous-phase inhibition is based on the formation of $SiCl_4$ and HCl that can be produced only at temperatures above 300°–500°C[1]:

350°–500°C:

1. $2SnCl_2 + nSi = 2Sn + (n - 1)Si + SiCl_4$
$SiCl_4 + 2H_2O = 4HCl + SiO_2$

410°C:

2. $2PbCl_2 + nSi = 2Pb + (n - 1)Si + SiCl_4$

280°–350°C:

3. $4CuCl + nSi = 4Cu + (n - 1)Si + SiCl_4$

300°C:

4. $2CaCl_2 + nSi = 2Ca + (n - 1)Si + SiCl_4$

400°C:

5. $4FeCl_3 + nSi = 4Sn + (n - 1)Si + 3SiCl_4$

In this temperature region $SiCl_4$ can depress the combustion of polymers in gaseous phase and also in the solid one. In solid phase $SiCl_4$ may react as "carbonizator" to produce a char. In gaseous phase $SiCl_4$ and HCl are the inhibitors of radical chain reaction of propagation in flame zone.

We have chosen the SI-system with $SnCl_2$ for polyolephynes (polypropylene) because of the wide temperature range of reaction 350°–500°C. Presumably the same temperature range may be encountered in solid phase of polymer during combustion.

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EXPERIMENTAL

Materials

The polymer used in this work was polypropylene, isotactic were supplied by Scientific Polymer Products, Inc., USA. The inorganic additives were, Stannous Chloride A.C.S. (Fisher Sci. Comp.), Sodium Borate, A.C.S. ($Na_2B_4O_7*10H_2O$, Fisher Sci. Comp.), Silicon, metal, 325 mesh, 99% (Aldrich Co.).

Preparation of Samples, Incorporation of Additives

Inorganic additives were mixed with polypropylene powder in laboratory blender. The samples were prepared by press moulding at temperatures 120–140°C during 4 minutes.

Cone calorimeter tests of the polymer samples, as discs (radius 35 mm), were carried out at 20, 30, 35 and 50 kW/m^2. Each specimen was wrapped in aluminum foil and only the upper face was exposed to the radiant heater.

RESULTS AND DISCUSSION

The cone data (20 kW/m^2) of polypropylene with Si-composition are given in Table I and Figures 1–6. The sodium borate (5 and 2% by wt.) was incorporated into these systems as intumescent agent. The addition of 10% wt. of silicon significantly depresses the flammability of polypropylene (Figure 1). However, the incorporation only 2% wt. of $SnCl_2$ has increased the ignition time delay in two times due to inhibition of combustion in gaseous phase by $SiCl_4$ and HCl.

The set of Cone tests was carried out at heat flux 35 kW/m^2 (Table II, Figures 7–11). The flame retardant composition included 3% wt. of Si and 2% of $SnCl_2$. All Cone results indicate an improvement of fire resistance of Si-polypropylene composition in comparison with pure polypropylene (Figures 7–11).

CONCLUSIONS

(1) The SI-system for polypropylene significantly suppressed the combustion of polypropylene. HCl and $SiCl_4$ inhibit the combustion in a gas phase and dramatically decrease the ignition time delay from 217 sec. to 394 sec. for polypropylene (20 kW/m^2, Table).

(2) Silicon-tin chloride polymer composition takes the "transition" place in the ecological scale of safety for flame retardants because of high temperature emanation of HCl and $SiCl_4$. However, these reactions take place only at temperatures above 300°C. While, at temperatures below 300°C SI-system is highly environmentally safe.

Reference

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