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### Fire Resistance Study of PP Thermoplastic Composites with Particulate Reinforcements and Br Flame Retardants

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가  
가  
가 85.34 μm, 33.93 μm, 가 18.51 μm 가  
(LOI, ASTM D2863) (ASTM E1354,  
ISO 5660) TGA  
DBDPO (M - HRR) > >  
가 가 2 DBDPO  
(OM) (SEM)

ABSTRACT : The fire resistance of particulate polypropylene composite systems were investigated by using various reinforced particles such as zeolite, talc, CaCO<sub>3</sub> particles. In this study, The effect of particle size on the thermal properties of composite and the effect of reinforced particles on the fire resistance were studied. The inorganic reinforced particles used in this study were recycled zeolite(average particle diameter=85.34 μm), CaCO<sub>3</sub> (33.93 μm), and talc(18.51 μm). The fire resistance of composite systems was thoroughly examined by measuring limited oxygen index (LOI, ASTM D2863) and cone calorimetry(ASTM E1354, ISO 5660). Thermal stability of composite systems was thoroughly examined by measuring TGA. The flame retardants (DBDPO) and reinforced particles reduce the maximum heat release rate (M - HRR) in the order of Talc > CaCO<sub>3</sub> > recycled Zeolite. Comparing the cone calorimetry experimental results of the particle reinforced polymer composite system exhibited twice higher efficiency than DBDPO in polypropylene systems, and the LOI also showed similar trends to the cone calorimetry experiments. The optical and scanning electron microscopy techniques were used to investigate the composites ash layer and the core fracture surfaces in the burning process. The reinforcing inorganic particles seemed to accumulate at the surface of ash layer, and subsequently intercept the oxygen transport and heat transfer into the core area.

Keywords : fire resistance, cone calorimetry, LOI, flame retardants(DBDPO), thermal stability, reinforced particles, recycled zeolite, CaCO<sub>3</sub>, talc.

(Br)

, 가

<sup>1-3</sup>

(GF), (CF),

가

가  
(virgin polypropylene, VPP)  
(M<sub>n</sub>) 37000, (M<sub>w</sub>) 57000  
( ) PP - 164(MFI=3.6 g/10 min)  
(recycled polypropylene, RPP)  
가 melt flow index (MFI)=  
3.4 g/10 min <sup>10</sup>  
가 ( )

(Br) 42%

가

(decabromodiphenyl oxide, DBDPO)

가

(Malvern

Instruments Ltd.)

85.34, 33.93, 18.51 μm

<sup>4-9</sup>

가

(PP)

(zeolite), (talc), (CaCO<sub>3</sub>)

가

(RPP), (DBDPO) 75 ,

85 95 kPa 24

(decabromodiphenyl oxide,

wt(%)

phr

DBDPO) 가

가

가

**Table 1. Sample Features Particle Reinforced Polymer Composites Containing Flame Retardants**

material sample	VPP wt(%)	RPP wt(%)	zeolite (%)	talc (%)	CaCO <sub>3</sub> (%)	DBDPO (phr)
VD-0	100					0
VD-9	100					9
VZD-737	70		30			7
VZD-739	70		30			9
VZD-7312	70		30			12
VZD-7314	70		30			14
VTD-739	70			30		9
VTD-7314	70			30		14
VCD-739	70				30	9
VCD-7314	70				30	14
RZD-739		70	30			9
RZD-7314		70	30			14

DBDPO

(char)

2

, SEM

가 (Brabender Co. Ltd) 235 250 , 30 rpm 가  
 230 245 , 30 55 MPa (FANUC. Co. Ltd.)  
 (100 × 100 × 5 mm) 가

Table 1

TGA(Thermal Gravimetry Analysis) : DBDPO 가 VPP

TA Instruments Ltd. TGA - 2050 TGA  
 10 /min, 11  
 12 mg N<sub>2</sub> , Air (LOI) :  
 ASTM D 2863 - 87

(150 × 7 × 3 mm) 가  
 SUGA ( )  
 11 - 13

$$LOI = \frac{[O_2]}{[N_2] + [O_2]} \times 100$$

[O<sub>2</sub>] : volumetric flow rate of oxygen (mm<sup>3</sup>/s)  
 [N<sub>2</sub>] : volumetric flow rate of nitrogen (mm<sup>3</sup>/s)

: (Fire Testing Technology Ltd.) , heat flux 25, 50, 75 kW/m<sup>2</sup> flow rate 0.024 m<sup>3</sup>/s ASTM E1354, ISO 5660  
 (HRR), (M - HRR), (TTI), (EHC), (THR), (CO) (CO<sub>2</sub>)  
 14 - 18

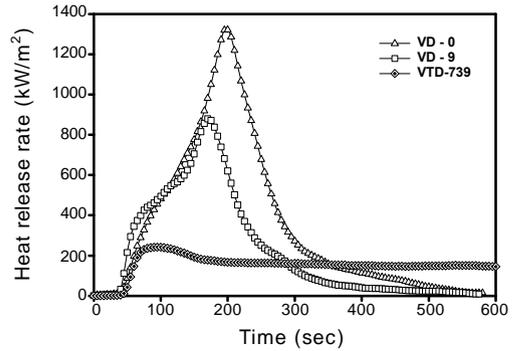


Figure 1. Heat release rate versus time for particle reinforced polymer composites of VPP/DBDPO/talc (50 kW/m<sup>2</sup>).

(Heat Release Rate, HRR) : Figure 1

DBDPO 가 가 HRR (maximum heat release rate, M - HRR)  
 (VD - 0) 1322 kW/m<sup>2</sup> , 가 가 (VZD - 739)가 404 kW/m<sup>2</sup>, (VCD - 739) 400 kW/m<sup>2</sup>  
 (VTD - 739) 241 kW/m<sup>2</sup>  
 5 가

가 2 4 HRR (Br , Cl )

10 Figure 1 HRR 가 flashover 19 - 21 HRR

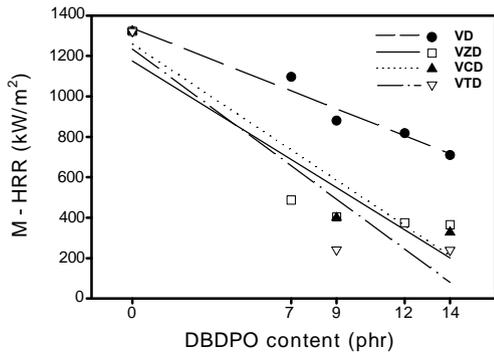
가 가 가 sintering

가 가 HRR >

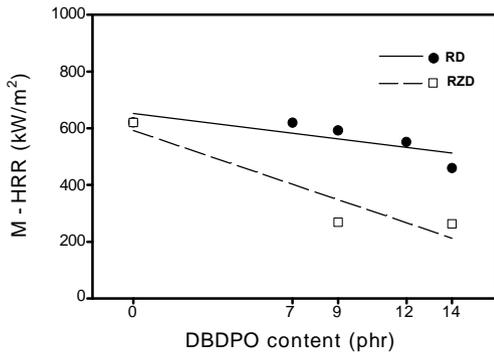
(Br)

**Table 2. Cone Calorimeter Data for Particle Reinforced Polymer Composites Containing Flame Retardants (TTI, M-HRR, A-HRR, THR, TTI/M-HRR, M-EHC, A-EHC, Char Formation(%))**

item (unit) sample	TTI (sec)	M - HRR (kW/m <sup>2</sup> )	A - HRR (kW/m <sup>2</sup> )	THR (MJ/m <sup>2</sup> )	TTI/M - HRR (s · m <sup>2</sup> /kW)	M - EHC (MJ/kg)	A - EHC (MJ/kg)	char formation (%)
VD - 0	37	1322	369	199.4	0.028	81.1	39.6	4.9
VD - 9	42	879	237	152.4	0.048	67.5	29.4	13.4
VZD - 737	22	487	235	109.7	0.045	44.5	28.6	35.6
VZD - 739	23	404	206	102.0	0.057	42.1	26.4	35.6
VZD - 7312	32	375	219	103.8	0.085	42.7	24.9	37.9
VZD - 7314	43	365	185	101.4	0.118	41.1	23.6	42.5
VTD - 739	46	241	130	129.2	0.191	49.2	25.7	34.4
VTD - 7314	57	239	123	121.5	0.238	44.8	25.5	35.5
VCD - 739	46	400	241	126.7	0.115	47.7	29.9	33.0
VCD - 7314	51	328	168	113.0	0.155	39.8	27.1	41.2
RZD - 739	40	269	187	97.7	0.149	54.0	25.8	41.5
RZD - 7314	49	263	149	92.6	0.186	37.8	24.0	50.1



(a)



(b)

**Figure 2. Efficiency of M - HRR versus various particle reinforced polymer composites of (a) VPP/reinforced particles/DBDPO and (b) RPP/reinforced particle/DBDPO (50 kW/m<sup>2</sup>).**

Table 2  
(M - HRR),  
(A - HRR)  
가 M - HRR A - HRR 가  
Figure 2 M - HRR  
DBDPO  
가  
(a) DBDPO 가  
1% 가 flashover M - HRR  
44.37 kW/m<sup>2</sup> · phr,  
69.54 kW/m<sup>2</sup> · phr,  
74.75 kW/m<sup>2</sup> · phr 82.45 kW/m<sup>2</sup> · phr  
가 2 가  
(RPP)  
가  
3 Figure 2 (b)  
(char formation)

(Time to Ignition, TTI) : Table 2  
 가 가 TTI  
 TTI , TTI (retardant effect)  
 18,22-24 (VZD)  
 9 phr  
 TTI (hole)

Figure 1 HRR  
 가  
 가 TTI (VTD) 가

Table 2 TTI M - HRR  
 (TTI/M - HRR)  
 가 가  
 flashover  
 가

(Total Heat Release, THR) : Table 2  
 THR , 가 가 THR  
 가

(Effective Heat of Combustion, EHC) :  
 Figure 3 (VD - 9)  
 가 가 (VTD - 739)

EHC  
 DBDPO 가  
 가  
 가  
 가  
 가  
 (void) , char  
 EHC

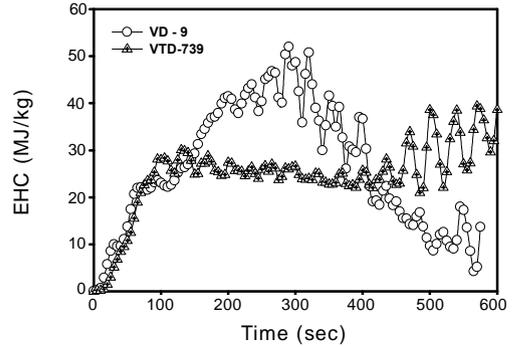


Figure 3. Effective heat of combustion versus time for particle reinforced polymer composites of VPP/DBDPO/Talc (50 kW/m<sup>2</sup>).

Table 2 (maximum effective heat of combustion, M - EHC)  
 (average effective heat of combustion, A - EHC)

가 EHC  
 Table 2  
 (%) 가  
 가 char

M - EHC  
 , Figure 4  
 DBDPO

(a)  
 가 2.22 MJ/kg · phr,  
 가 2.85 MJ/kg · phr,  
 3.04 MJ/kg · phr 2.70 MJ/kg · phr

(b)  
 가  
 가 가  
 Figure 5 CO CO<sub>2</sub>

(CO and CO<sub>2</sub> Production Rate) : Figure 5

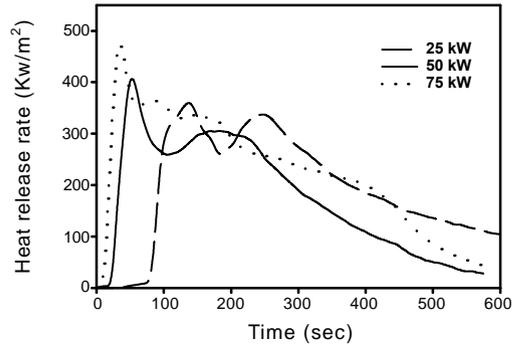


**Table 3. Efficiency of Cone Calorimeter Data for Particle Reinforced Polymer Composites Containing Flame Retardants**

sample	item (unit)	M - HRR (kW/m <sup>2</sup> · phr)	M - EHC (MJ/kg · phr)	LOI (%/phr)
VPP+DBDPO		- 44.37	- 2.22	0.48
VPP+Zeolite+DBDPO		- 69.54	- 2.85	0.59
VPP+CaCO <sub>3</sub> +DBDPO		- 74.75	- 3.04	0.62
VPP+Talc+DBDPO		- 82.45	- 2.70	0.74
RPP+DBDPO		- 9.94	- 2.04	0.44
RPP+Zeolite+DBDPO		- 27.11	- 2.48	0.67

**Table 4. Cone Calorimeter Data for Virgin PP/Zeolite/DBDPO (70/30/9) (heat flux - 25, 50, 75 kW/m<sup>2</sup>)**

item(unit)	heat flux (kW)		
	25 kW	50 kW	75 kW
TTI (sec)	77	23	10
M - HRR (kW/m <sup>2</sup> )	359	404	473
THR (MJ/m <sup>2</sup> )	168.9	131.6	102.0
TTI/M - HRR (s · m <sup>2</sup> /kW)	0.214	0.057	0.021
M - EHC (MJ/kg)	42.1	52.9	59.9
Max. [CO]/[CO <sub>2</sub> ] char formation (%)	0.167	0.159	0.127
	38.4	37.2	35.6



**Figure 6.** Heat release rate versus time for various heat flux (25, 50, 75 kW/m<sup>2</sup>) of VPP/zeolite/DBDPO (70/30/9).

739) 가

Table 4  
HRR TTI  
Figure 6  
heat flux  
75 kW 가  
25 kW 가  
75 kW 가  
char crack  
, 50, 25 kW  
char crack  
char crack  
75 kW  
char crack

25 kW 가  
50 kW char crack 가 crack

가 가<sup>26</sup>

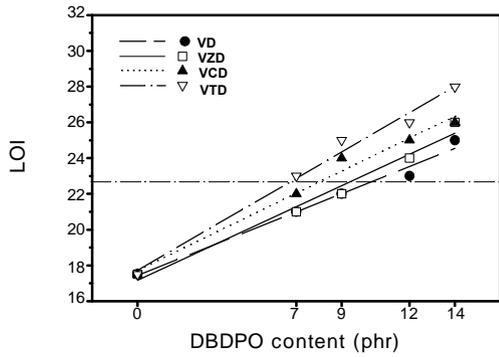
Table 4  
(TTI)

25 kW 가  
<sup>26,27</sup>  
(LOI) : Figure 7(a)  
가

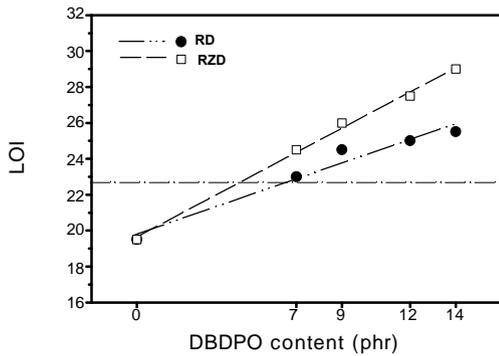
가 LOI  
LOI 가  
LOI가 가  
LOI  
LOI가  
(UL94 V2) LOI  
25 가  
LOI  
<sup>27</sup>  
가

DBDPO 가 0.48 LOI(%)/  
phr, 0.59 LOI(%)/phr,

(Br)



(a)



(b)

**Figure 7.** Efficiency of limited oxygen index versus various particle reinforced polymer composites of (a) VPP/reinforced particles/DBDPO and (b) RPP/reinforced particle/DBDPO (50 kW/m<sup>2</sup>).

0.62 LOI(%) / phr      0.74 LOI(%) / phr  
DBDPO      가

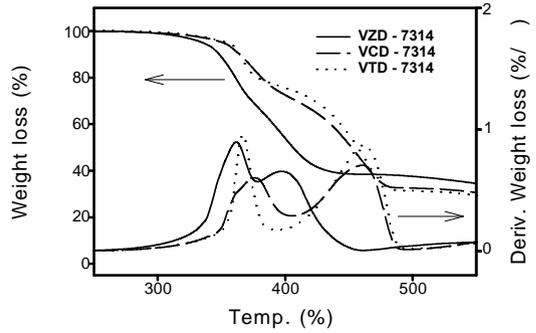
가      Table 3

(TGA)      Figure 8

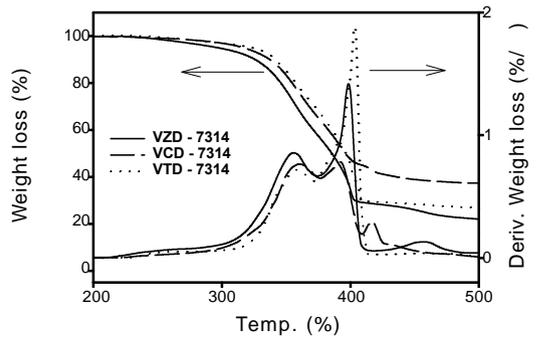
가      가  
N<sub>2</sub>      air      PP  
가      가

(VD)      가  
423      Figure 8 (a)      VZD - 7314      398  
, VCD - 7314      461      , VTD - 7314

463



(a)



(b)

**Figure 8.** TGA thermogram of VPP/Reinforced Particles/DBDPO in (a) N<sub>2</sub> purge and (b) air conditions.

(b)      VD      가 316      VZD - 7314      398  
, VCD - 7314      392      , VTD - 7314      402.5

<sup>10,18</sup>

가      가      가  
가      ,      (Br · )

(char)

<sup>28-31</sup>

VPP가 3.3 kg<sub>f</sub> · cm/cm<sup>2</sup>  
(VTD - 739)가      가      7.99 kg<sub>f</sub> · cm/cm<sup>2</sup>

2 가  
 VPP가 2800 MPa  
 (VTD - 739)가 가 4473 MPa  
 가 가  
 가 가  
 가 가  
 가 PP  
 가 가  
 가 가  
 VPP DBDPO 가  
 가 가  
 가 가  
 HRR 2 4  
 , TTI 가  
 14 phr 가  
 25 가  
 9 phr 가  
 25  
 TGA DBDPO 40  
 가 VPP  
 TGA 가 가 가  
 가 가  
 , PP가 가  
 가

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