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Predicting Materials' Ease of Combustion: Development of a Simple Test Method

By Maria I. De Rosa and Charles D. Litton

UNITED STATES DEPARTMENT OF THE INTERIOR

BUREAU OF MINES

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UNIT OF MEASURE ABBREVIATIONS USED IN THIS REPORT

°C	degree Celsius	L/min	liter per minute
cm	centimeter	min	minute
cm^2/p	square centimeter per particle	μm	micrometer
g	gram	p/cm^3	particle per cubic centimeter
in	inch	pct	percent
L	liter	s	second

PREDICTING MATERIALS' EASE OF COMBUSTION: DEVELOPMENT OF A SIMPLE TEST METHOD

By Maria I. De Rosa[1] and Charles D. Litton[2]

ABSTRACT

The U.S. Bureau of Mines conducted experiments for predicting materials' ease of combustion (smoldering onset, smoldering, flaming, and decomposition rates) by means of submicrometer smoke particle characteristics for the development of a simple test method. The experiments were carried out in an approximately 20-L furnace, with a 10-L/min airflow through the furnace for a 14-min duration, at set furnace temperatures of 150°, 250°, and 1,000° C.

The variables studied as a function of time were the onset time of smoke particles, time and duration of maximum smoke particle generation, average concentration of smoke particles, and particles' average diameter, mass weight loss, and furnace temperatures. Results show that the onset time of smoke particles is predictive of materials' ease of smoldering, and the time of maximum smoke particle generation and its duration, coupled with mass weight loss, are predictive of materials' ease of smoldering and flaming (depending on the experimental temperatures), and decomposition rates.

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INTRODUCTION

In 1984, the U.S. Bureau of Mines, within its mission to create and maintain safety in the mines and, specifically, to assess the hazard of mine materials during fire, initiated a series of experiments to characterize smoke particles and primary gas toxicities evolved during the combustion of mine materials. It was found (*1-8*)[3] that the product ($d_g n_o$) of smoke particles' average diameter (d_g) and particles' average concentration (n_o) varied with the main toxicities evolved during the combustion of chlorine-containing (hydrogen chloride and carbon monoxide) and nitrogen-containing (hydrogen cyanide and carbon monoxide) materials. The earlier and faster these types of materials burnt, the earlier and faster the main toxicities evolved, and the larger (nitrogen-containing materials) or smaller (chlorine-containing materials) the $d_g n_o$ values were.

Could a simple test method be developed for the prediction of materials' most important fire parameter such as materials' ease of combustion? In the present study, the Bureau carried out experiments to develop a simple test method for the evaluation of these fire parameters by studying the onset time of smoke particles (T_o), and the time of maximum smoke particle generation (Tmax) and its duration (T_d).

BACKGROUND

The first line of defense of any material against fire is its resistance to combustion. Many tests have been developed to examine materials' fire parameters. These tests determined the time at which temperatures shoot up [Setchkin test (*9*)]; the time to ignition [Fristrom test (*10*) and intermittent flame test (*11*)], the time to ignition by gradually varying the oxygen concentration [limiting oxygen index test (*12*) and American Standard Test Method (ASTM) D2863 (*13*)], the rates of heat release in a controlled atmosphere [calorimetric method (14)], the flame spread [2-ft-tunnel test (15)], and finally, the burning rates [opposed flow diffusion flame test (16-17)].

The majority of these tests, however, allow for arbitrary observations and conclusions; other tests allow extremely small variances between the passing and failing ratings; above all, numerous and complicated tests are needed for the evaluation of a few selected fire parameters.

EXPERIMENTAL SYSTEM

The system (fig. 1) consists of an approximately 20-L furnace whose temperature during experiments rises automatically from ambient (fig. 2) at a rate depending on the set furnace temperature, which ranges from 100° to 1,200° C. The experimental temperatures are monitored continuously with type K thermocouples connected to a strip-chart recorder. A universal load cell, located under the furnace floor and contacted by a sample-cup pedestal, transmits voltages of sample mass weight loss via a bridge amplifier to another strip-chart recorder. A vacuum pump draws ambient air (10 L/min) continuously into the furnace via an opening on the furnace door and sends the resulting combustion air through a quartz tubing to the submicrometer particle detector analyzer (SPDA) (*18*) and the exhaust hood.

Flowmeters, installed between the pump outlet and the SPDA, provide visual flow indication. Data of furnace temperature, sample weight loss, and SPDA voltages, used to compute n_o and d_g, are continuously acquired, mathematically treated, logged, filed, displayed, and plotted by means of a miniframe computer via a laboratory-based real-time data acquisition system.

EXPERIMENTAL PROCEDURE

Three sets of experiments (eight experiments in the set; each experiment repeated three times) were performed at set furnace temperatures of 150° (unsustained smoldering stage of combustion), 250° (sustained smoldering stage of combustion), and 1,000° C (flaming stage of combustion) (fig. 2) for a 14-min duration with 1-g samples of polyvinyl chloride (PVC) (P1), neoprene (N1), and styrene-butadiene rubber (SBR) (S1) mine conveyor belts; PVC (B1), fiberglass (F1), and jute (J1) mine brattices; coal dust (C1); and wood shavings (W1). A list of the materials is reported in table 1. A 1-g sample was placed in a 2.5-cm-diameter sample cup, inserted in a quartz (70-cm)

[3] Italic numbers in parentheses refer to items in the list of references at the end of this report.

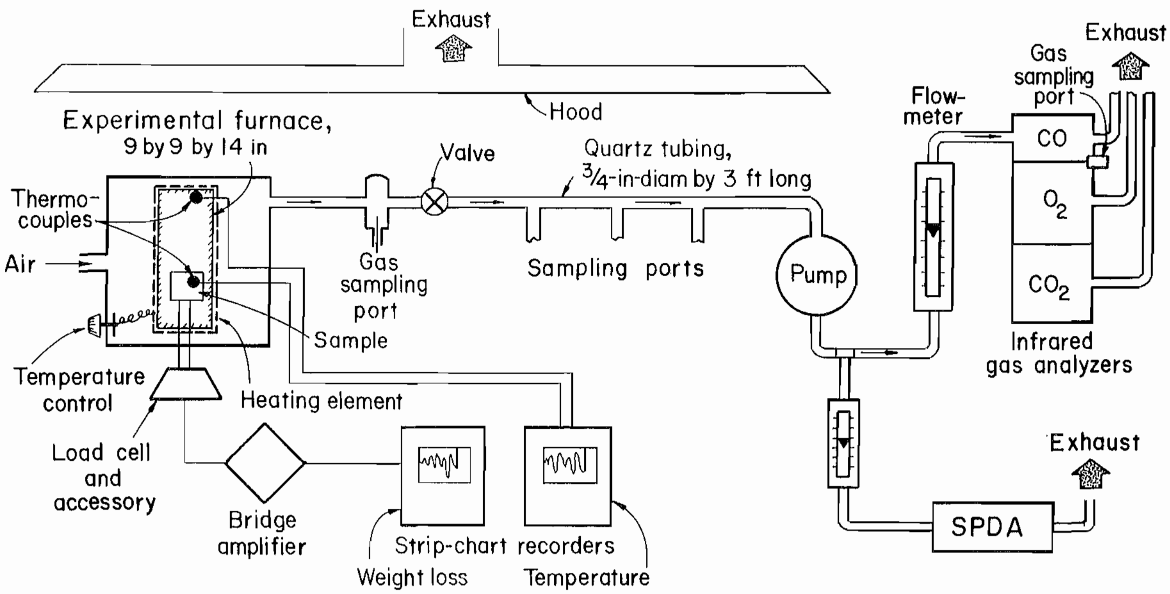

Figure 1.—Experimental system. (SPDA = submicrometer particle detector analyzer)

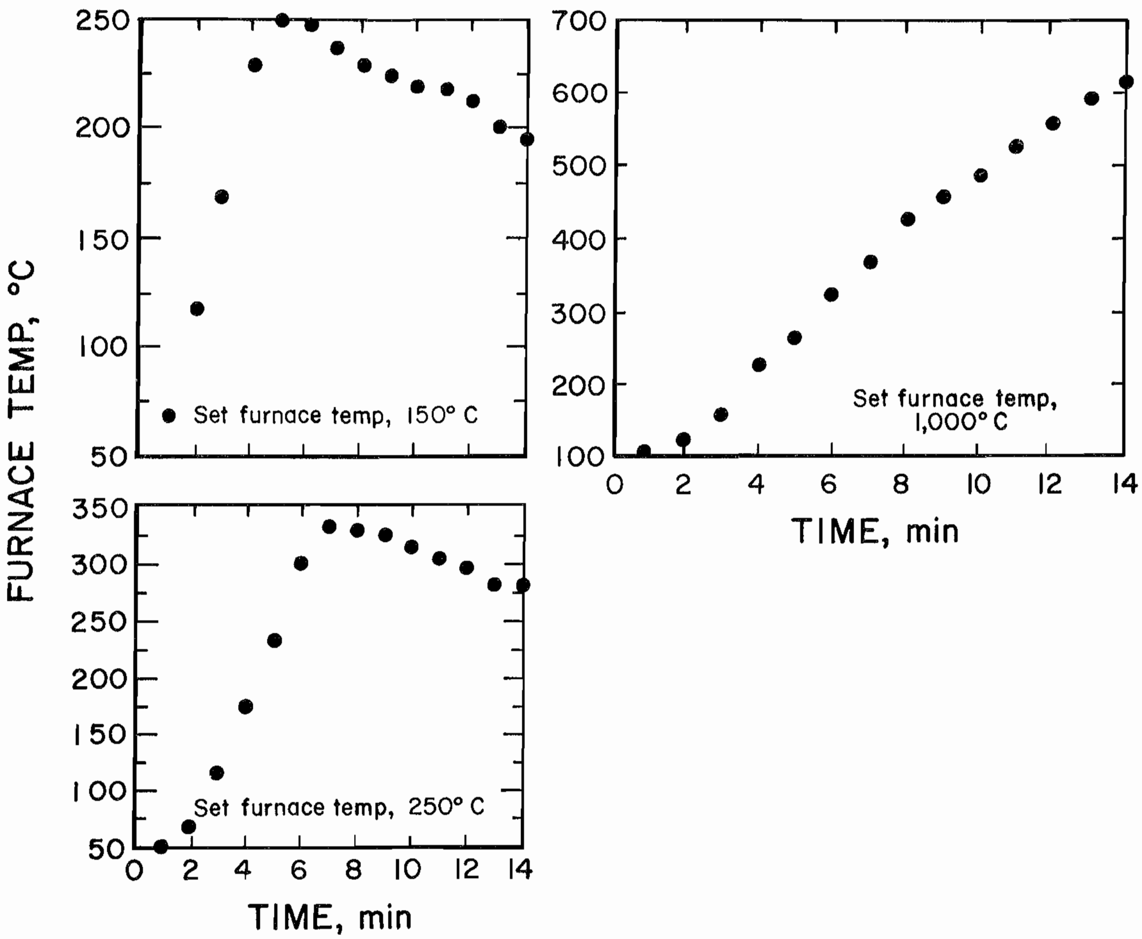

Figure 2.—Furnace temperature versus time at set furnace temperatures of 150°, 250°, and 1,000° C.

pedestal, and the furnace was set at the desired temperature with a 10-L/min airflow through the furnace for a 14-min duration. The resulting combustion air was directed (1.6 L/min) into the SPDA, and the remaining air was directed under the exhaust hood. Under each set furnace temperature, the variables studied were T_o (minutes), smoke particles of average concentration (particles per cubic centimeter) and d_g (centimeters reported as micrometers), at onset time; Tmax (minutes) and T_d (minutes), smoke particles of average concentration (Tmax n_o) (particles per cubic centimenter) and d_g (centimeters reported as micrometers), at maximum generation time. The data of n_o and d_g, calculated at 10-s intervals, were reported as average for each minute. Other variables studied were the total mass weight loss (grams) and furnace temperatures (degrees Celsius). The d_g is obtained by calculating $d_g n_o$ (particles per square centimeter) from the ratio of the SPDA experimental (with smoke) and initial (without smoke) current output (Ie/Io), following the relationships in equation 1 (see also figure 3).

$$Ie/Io = 1/(K\, d_g n_o)\,(1-\exp^{(-K\, d_g n_o)}), \qquad (1)$$

where K = charging constant (K 0.012, cm^2/p).

Once the $d_g n_o$ is determined, d_g can be obtained from equation 2:

$$d_g = (\exp^{Ic/d_g n_o - 1})/(Ic), \qquad (2)$$

where Ic = SPDA charged particle current,

and $d_g n_o \div d_g = n_o$.

Table 1.—Materials investigated

Material	Description	Cl_2, pct
Conveyor belt:		
PVC (P1) . . .	Polymer component is PVC with fillers.	23
Neoprene (N1)	Polymer component is neoprene rubber with fillers.	11
SBR (S1) . . .	Polystyrene-butadiene rubber treated for fire retardancy with chlorinated additives.	5
Brattice:		
PVC (B1) . . .	Polymer component is PVC with fillers.	28
Fiberglass (F1)	Fiberglass (<90 pct) fibers treated for fire retardancy with chlorinated additives.	5
Jute (J1) . . .	Jute fibers treated for fire retardancy with chlorinated additives.	5
Coal (C1)	Coal dust (200 mesh), Pittsburgh Seam.	0
Wood (W1)	Pine wood shavings, untreated.	0

PVC Polyvinyl chloride.
SBR Styrene-butadiene rubber.

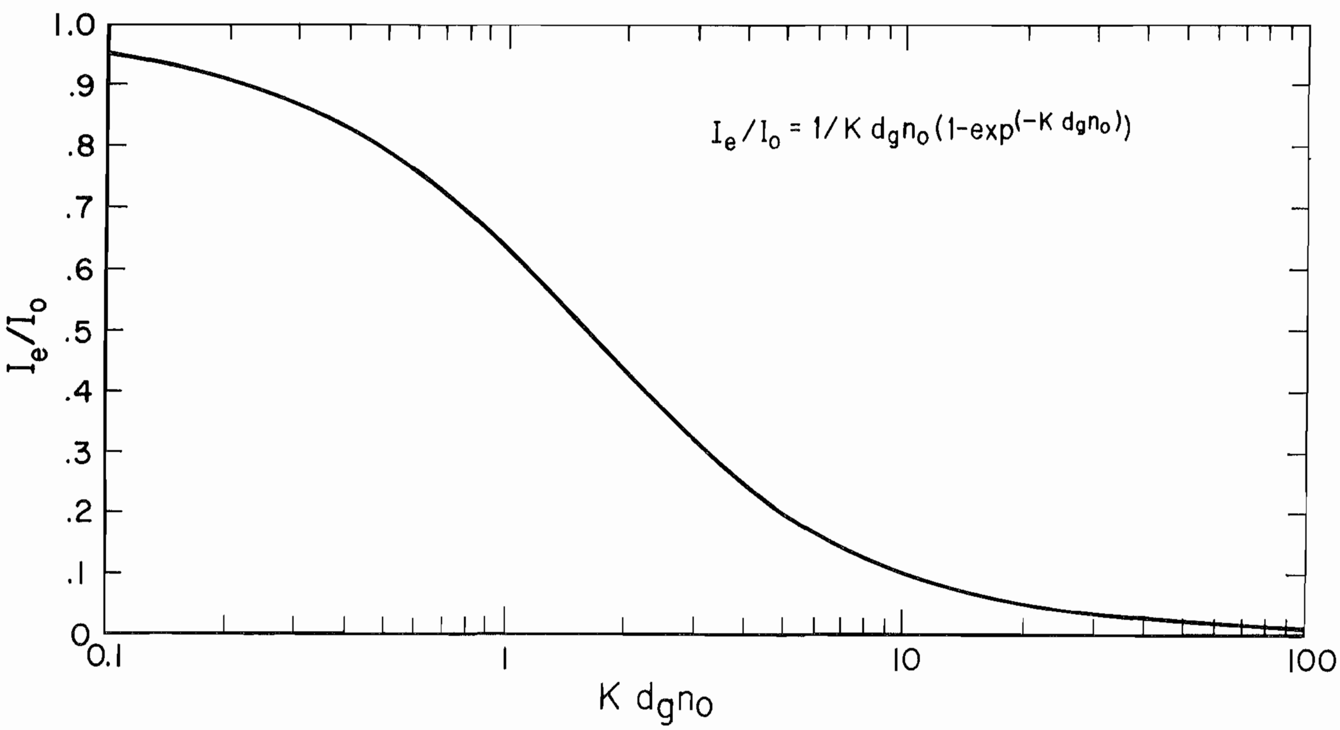

Figure 3.—Submicrometer particle detector analyzer (SPDA) current ratio (Ie/Io) as function of smoke particle diameter and concentration product ($d_g n_o$).

RESULTS AND DISCUSSION

At 150° C set furnace temperature (unsustained smoldering stage of combustion), all materials smoldered lightly for a very short period of time, yielding small particle concentrations. The PVC brattice, the PVC belt, and the wood samples, followed by the jute brattice and the SBR belt samples, yielded the earliest onset time of smoke particles, ranging between the sixth minute for the PVC materials and the wood, and the eighth minute for the other materials (table 2 and figures 4 and 5). The coal sample, followed by the fiberglass brattice and the neoprene belt samples, yielded the latest onset time, ranging between the 11th minute for the coal and the 9th minute for the fiberglass brattice and the neoprene belt.

Table 2.—Thermal decomposition data at set furnace temperatures of 150°, 250°, and 1,000° C. (Furnace airflow of 10-L/min for a 14-min duration).

Material[1]	At onset time of smoke particles				At time of maximum smoke particle generation					
	T_o, min	$T_o n_o$, 10^6 p/cm^3	d_g, µm	F temp, °C	Tmax, min	T_d, min	Tmax n_o, 10^6 p/cm^3	d_g, µm	F temp, °C	Total WL, g
					150° C					
Conveyor belt:										
PVC (P1) . . .	6	0.01	0.002	190	7-12	6	0.4 - 0.2	0.004-0.009	190-170	0.01
Neoprene (N1)	9	2.6	.003	183	10-12	3	3 - 4	.004- .006	179-170	.0001
SBR (S1) . . .	8	1.3	.001	187	9-12	4	3 - 4	.002- .003	183-170	.05
Brattice:										
PVC (B1) . . .	6	.01	.012	190	7-12	6	.03- .2	.015- .002	190-170	.05
Fiberglass (F1)	9	.01	.559	213	10-12	3	.3 - .8	1.25 - .18	180-170	.0001
Jute (J1) . . .	8	4.7	.001	187	9-12	4	20 - 25	.001- .001	187-170	.05
Coal (C1)	11	.01	.022	195	12	1	.05	.007	176	.0001
Wood (W1)	6	9	.001	190	7-12	6	10 - 3.5	.003- .001	187-179	.05
					250° C					
Conveyor belt:										
PVC (P1) . . .	5	0.12	0.001	257	6-14	9	0.6 - 4	0.001-0.014	294-277	0.08
Neoprene (N1)	8	2	.474	304	9-14	6	3 - 11	.004- .001	304-277	.02
SBR (S1) . . .	7	18	.005	304	8-14	7	27 -170	.004- .003	304-277	.15
Brattice:										
PVC (B1) . . .	5	.14	.776	172	6-14	9	.4 - 1	.271- .238	286-270	.45
Fiberglass (F1)	8	.1	.014	310	9-14	6	.5 - .7	.018- .003	298-270	.001
Jute (J1) . . .	7	41	.003	294	8-14	7	81 - 50	.004- .004	271-274	.42
Coal (C1)	10	.1	.022	272	11-14	4	.2 - 1.2	.021- .001	265-268	.03
Wood (W1)	5	11	.002	294	6-14	9	11 - 1	.010- .012	271-286	.32
					1,000° C					
Conveyor belt:										
PVC (P1) . . .	4	0.12	0.023	300	5-12	8	0.19-126	1.4 -0.004	307-565	0.99
Neoprene (N1)	7	10	.053	350	8-14	7	267 - 2	.020- .008	392-565	.99
SBR (S1) . . .	6	66	.341	307	7-12	6	140 -190	.060- .020	350-565	.99
Brattice:										
PVC (B1) . . .	4	.10	.284	228	5-10	6	27 - 31	.053- .062	286-520	.99
Fiberglass (F1)	7	.10	.001	355	8-12	5	7.5 - 29	.003- .003	398-578	.09
Jute (J1) . . .	6	1.6	.002	307	7-10	4	150 -115	.004- .004	331-550	.62
Coal (C1)	9	.1	.016	374	10-14	5	.3 - 10	.248- .052	414-555	.27
Wood (W1)	4	16	.002	238	5-10	6	230 - 86	.004- .003	286-550	.98

d_g	Smoke particles' average diameter at time of smoke particle onset and at time of maximum smoke particle generation.
F temp	Furnace temperature at time of smoke particle onset and at time of maximum smoke particle generation.
PVC	Polyvinyl chloride.
SBR	Styrene-butadiene rubber.
T_d	Duration time of maximum smoke particle generation.
Tmax	Time of maximum smoke particle generation.
Tmax n_o	Smoke particle concentration at maximum generation time.
T_o	Smoke particle onset time.
T_o n_o	Smoke particle concentration at onset time.
WL	Weight loss.

[1]1-g sample.

Also under the 150° C conditions, the PVC materials and the wood, followed by the jute brattice and the SBR belt samples, yielded the earliest time of maximum smoke particle generation, ranging between the seventh minute for the PVC materials and the wood, and the ninth minute for the jute brattice and the SBR belt (table 2 and figures 4 and 5), with a duration time of 6 min for the PVC brattice, the PVC belt, and the wood, and 4 min for the jute brattice and the SBR belt (table 2 and figure 4). Small but observable weight loss were observed for these materials, ranging between 5 pct for the brattices, the wood and the SBR belt, and 1 pct for the PVC belt. The coal, followed by the fiberglass brattice and the neoprene belt, yielded the latest time of maximum smoke particle generation, ranging between the 12th minute for the coal and the 10th minute for the fiberglass brattice and the neoprene belt, and the shortest duration time, ranging between 1 min for the coal and 3 min for the fiberglass brattice and the neoprene belt. Insignificant weight loss (0.01 pct) were observed for these materials.

At 250° C set furnace temperature (sustained smoldering stage of combustion), the PVC materials and the wood, followed by the jute brattice and the SBR belt, yielded the earliest onset time, ranging between the fifth minute for the PVC materials and the wood, and the seventh minute for the jute brattice and the SBR belt. The coal, followed by the fiberglass brattice and the neoprene belt, yielded the latest onset time, ranging between the 10th minute for the coal, and the 8th minute for the brattice and the belt (table 2 and figures 4 and 5).

Also under the 250° C conditions, the PVC materials and the wood, followed by the jute brattice and the SBR belt, yielded the earliest maximum generation time, ranging between the sixth minute for the PVC materials and the wood, and the eighth minute for the jute brattice and the SBR belt. These materials also yielded the longest

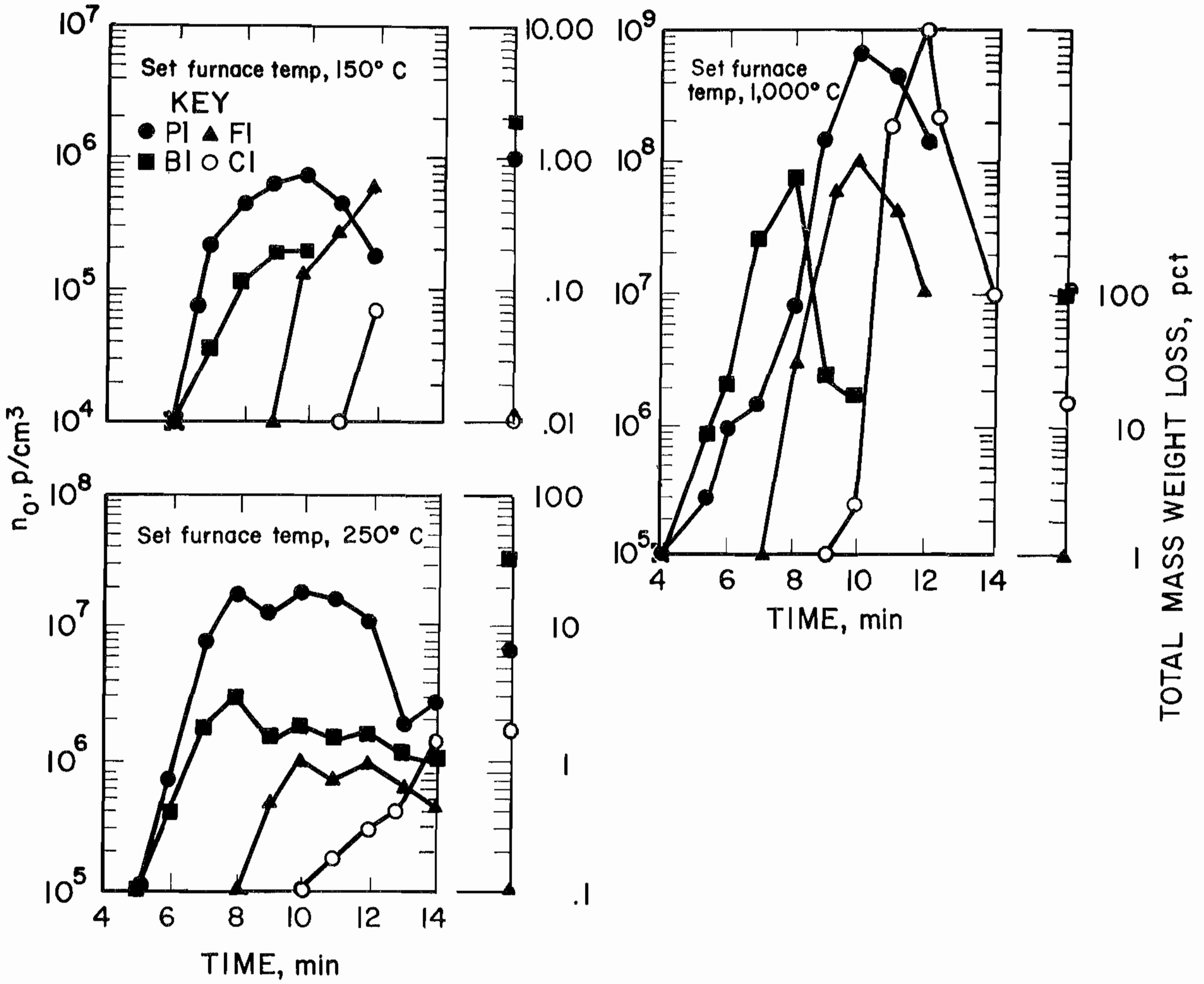

Figure 4.—Average concentration of smoke particles (n_0) and mass weight loss versus time at set furnace temperatures of 150°, 250°, and 1,000° C. Far right measurements taken at end of 14th minute. (P1 = polyvinyl chloride belt, B1 = polyvinyl chloride brattice, F1 = fiberglass brattice, C1 = coal)

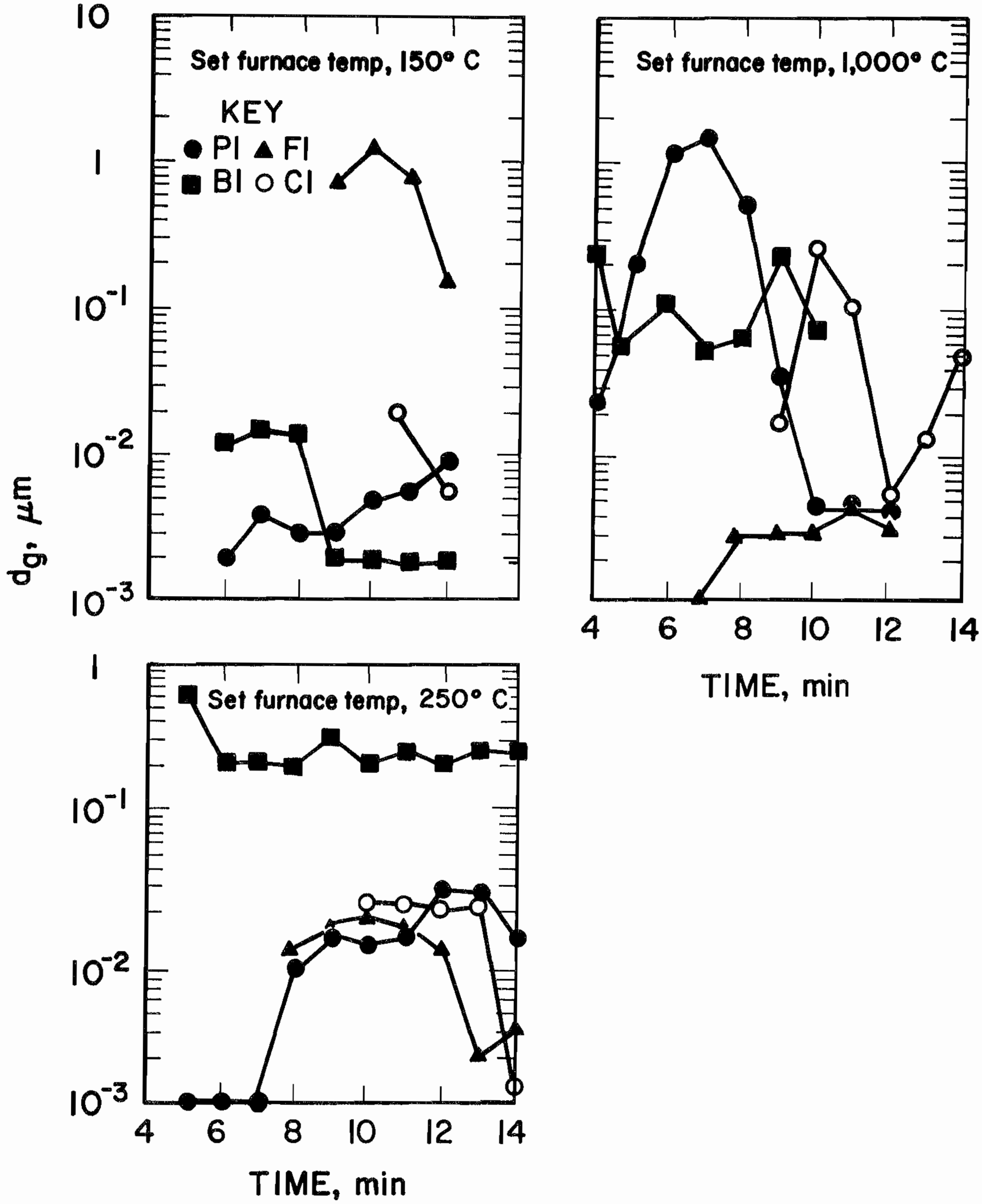

Figure 5.—Average diameter of smoke particles (d_g) versus time at set furnace temperatures of 150°, 250°, and 1,000° C. (P1 = polyvinyl chloride belt, B1 = polyvinyl chloride brattice, F1 = fiberglass brattice, C1 = coal)

duration time of 9 min for the PVC materials and the wood, and 7 min for the jute brattice and the SBR belt (table 2 and figure 4). Large weight losses were observed for these materials, ranging between ~45 pct for the brattices, 32 pct for the wood, 15 pct for the SBR belt, and 8 pct for the PVC belt. The coal, followed by the fiberglass brattice and the neoprene belt, yielded the latest maximum generation time, ranging between the 11th minute for the coal, and the 9th minute for the brattice and the belt. These materials also yielded the shortest duration time, ranging between 4 min for the coal, and 6 min for the brattice and the belt. Small weight loss were observed for these materials, ranging between 3 pct for the coal, 2 pct for the belt, and 0.1 pct for the brattice.

At 1,000° C set furnace temperature (flaming stage of combustion), the PVC materials and the wood, followed by the jute brattice and the SBR belt, yielded the earliest onset time, ranging between the fourth minute for the PVC materials and the wood, and the sixth minute for the brattice and the belt. The coal, followed by the fiberglass brattice and the neoprene belt, yielded the latest onset time, ranging between the ninth minute for the coal, and the seventh minute for the brattice and the belt (table 2 and figures 4 and 5).

Also under the 1,000° C conditions, the PVC materials and the wood, followed by the jute brattice and the SBR belt, yielded the earliest maximum generation time, ranging between the fifth minute for the PVC materials and the wood, and the seventh minute for the brattice and the belt; with a duration time ranging between 6 min for the PVC brattice, the wood, and the SBR belt (total weight loss of 100 pct); 8 min for the PVC belt (total weight loss of 100 pct); and 4 min for the jute brattice (total weight loss of 100 pct). Evidently, the short duration time is due to early, fast, and complete thermal decomposition of the samples (table 2 and figure 4). The coal, followed by the fiberglass brattice, yielded the latest maximum generation time, ranging between the 10th minute for the coal, and the 8th minute for the brattice; with a duration time of 5 min for the coal (total weight loss of <30 pct) and for the brattice (total weight loss of <1 pct). The short duration time for these latter materials is due, evidently, to incomplete thermal decomposition (coal), and to extremely low thermal decomposition (brattice). Surprisingly, the neoprene belt, yielding a maximum generation time at the eighth minute, with a duration of 7 min, underwent complete thermal decomposition (total weight loss of 100 pct).

CONCLUSIONS

The PVC brattice, the PVC belt, and the wood shavings samples, followed by the jute brattice and the SBR belt samples, decompose much earlier and at faster rates than the coal and the fiberglass brattice samples, at every set furnace temperature tested. The neoprene belt sample decomposes earlier, burning at faster rates, only at set furnace temperatures of 1,000° C.

During the unsustained smoldering stage of combustion (~200° C), the PVC belt, the wood shavings, the jute brattice, and the SBR belt samples yielded smoke particles earlier and for longer periods of time, undergoing small but measurable weight loss. On the contrary, the coal, followed by the fiberglass brattice and the neoprene belt, yielded smoke particles much later, for shorter periods of time, undergoing insignificant weight loss.

During the sustained smoldering stage of combustion (~300° C), the PVC materials and the wood, followed by the jute brattice and the SBR belt, yielded smoke particles earlier and for longer periods of time in large quantities, undergoing significant weight loss. On the contrary, the coal, followed by the fiberglass brattice and neoprene belt, yielded smoke particles later and for shorter periods of time in smaller quantities, undergoing small weight loss.

During the flaming stage of combustion (>400° C), the PVC materials', the wood, the jute brattice, and the SBR belt samples, and surprisingly, the neoprene belt sample yielded smoke particles at the earliest time, in extremely large quantities, undergoing complete thermal decomposition in a very short time. On the contrary, the coal and the fiberglass brattice yielded smoke particles later and for shorter periods of time, undergoing small weight loss.

In conclusion, the onset time of smoke particles is predictive of materials' smoldering onset, and the time of maximum smoke particle generation and its duration, coupled with mass weight loss, are predictive of materials' ease of smoldering and flaming (depending on the experimental temperatures), and decomposition rates. This prediction may allow the development of a simple, inexpensive, and efficient test method for the evaluation of materials' most important fire hazard parameters.

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