METHODS FOR ASSESSING THE FIRE PERFORMANCE OF PHENOLIC RESINS AND COMPOSITES

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ABSTRACT

An evaluation has been made of the behaviour of selected phenolic resins and glass-reinforced phenolic composites in a number of fire test procedures which ranged from bench-scale to full-scale and assessed a number of fire hazard characteristics. It was concluded that all the tests had some deficiencies but that the ASTM E-1354 Cone Calorimeter was most suitable for use in research and development of new materials and that the ASTM Room Fire Test had good potential for assessing 'real-life' fire performance.

1. INTRODUCTION

As part of a research project to develop phenolic composites with improved properties, it was necessary to assess the fire properties of both glass-reinforced phenolic composites and the plastics materials used in the composites. Test procedures were required for both developmental materials where only limited quantities were available and for composites manufactured on a pilot-scale where a 'realistic' full-scale evaluation was most appropriate.

Phenolic plastics generally exhibit superior fire behaviour and thus the tests used needed to provide sufficiently severe conditions, either in the form of the heat input of the ignition source or an applied radiant heat load. The procedures used were chosen on the basis of a literature search of past work on the fire behaviour of phenolic materials, test procedures currently used in Australia for materials and products used in buildings, and commonly used international procedures.

This paper discusses the test procedures used, the results obtained for selected materials and composites, and the utility of the tests for the various purposes.

2. EXPERIMENTAL

2.1 Materials

The phenolic resins were prepared from Cellobond J2024L and Phencat 10 (6% w/w). For the composites, the glass fibre used was four sheets of AC 200 chopped strand mat (450 g/m^2) plus two outer sheets of Regina glass tissue (30 g/m^2).

2.2 Specimen preparation

Specimens were either hand-batched or prepared by a commercial fabricator, as described in Table 1. The hand-batched plastics were moulded between two glass plates 300 x 300 mm, whilst the glass-reinforced composites were laid up on a single plate. The commercially fabricated specimens were laid up on a single glass sheet 2440 x 1220 mm. Resins and composites were allowed to gel at room temperature for 1 hour and then cured at 60°C for 2 hours, before being cut to the sizes required as specimens for each test. The cured sheets were nominally 5 mm thick.

Table 1. Composition and preparation of specimens

Specimen	Composition	Preparation
Phenolic 3U	Resin: Cellobond J2024L/Phencat 10 Glass fibre: nil	Hand-batched
Phenolic 3R	Resin: Cellobond J2024L/Phencat 10 Resin to glass ratio: 2:1	Hand-batched
Phenolic 3F	Resin: Cellobond J2024L/Phencat 10 Resin to glass ratio: 2:1 Density: 1250 kg/m^3	Commercially fabricated

2.3 Test procedures

The following procedures were selected for evaluation, although not all materials were submitted to all tests (see Table 2).

Table 2. Tests performed

Test method	Phenolic 3U	Phenolic 3R	Phenolic 3F
AS 1530.3 Australian Radiant Panel			X
ASTM E-662 Smoke Chamber		X	X
ISO 5657 Ignitability			X
ASTM E-1354 Cone Calorimeter	X	X	X
Room Fire Test[a]	–	–	X

[a] Specimens and burner in accordance with ASTM method[(6)]; burner program in accordance with the ISO 9705 method[(7)].

(a) AS 1530, Part 3[(1)]: 'Simultaneous Determination of Ignitability, Flame Propagation, Heat Release and Smoke Release'. The specimen is held vertically in a plane parallel to a radiant panel and the specimen moves towards the heater in steps over a period of 20 minutes or until ignition, induced by a non-impinging pilot flame, occurs. The fire performance parameters related to flame propagation, heat release and smoke release are then evaluated.

(b) ASTM E-662–83[(2)]: 'Standard Test Method for Specific Optical Density of Smoke Generated by Solid Materials'. Specimens were tested in both non-flaming and flaming modes.

(c) ISO 5657[(3)]: 'Fire Tests – Reaction to Fire – Ignitability of Building Products'. This method has been adopted in Australia as AS 1530, Part 5[(4)]; the methods are technically identical. The baseboards used in this project were fibre-reinforced cement with a thickness of 4.5 mm and density of 1300 kg/m^3, and hence were thinner but more dense than those specified in the Standard.

(d) ASTM E-1354–90[5]: 'Standard Test Method for Heat and Visible Smoke Release Rate for Materials and Products Using an Oxygen Consumption Calorimeter' commonly known as the 'Cone Calorimeter'. Specimens were tested in the horizontal configuration; using the retainer frame and wire grid.

(e) ASTM proposed fire test[6]: The method used was as specified except that the burner program was that in ISO–9705[7]. Specimens were mounted on walls only as specified.

3. RESULTS AND DISCUSSION

3.1 Previous work

Phenolic resins and glass-reinforced phenolic composites have been shown to be among the better performers when their fire behaviour is compared to that of other glass-reinforced plastics[8–15]. For instance, when assessed in the ASTM E-662 Smoke Chamber, in the flaming mode, some glass-reinforced phenolics gave maximum specific optical densities of about half those of some glass-reinforced polyesters[12,13,14] and, in the non-flaming mode, the glass-reinforced phenolic gave a maximum specific optical density of only 1/200th of the value for the glass-reinforced polyesters[13]. When tested in the BS 6853 3 m cube smoke test[16], a glass-reinforced phenolic obtained smoke levels of approximately 1/100th of that obtained by a glass-reinforced polyester[14].

In the Cone Calorimeter, glass-reinforced phenolics have performed better than glass-reinforced polyesters or epoxies with respect to ignition time, average heat release rate over 180 seconds and amount of smoke produced[8], and better than glass-reinforced polyesters and vinylesters in respect to ignition time, peak heat release rate and, with the exception of one vinylester at low irradiance, amount of smoke produced[9].

In other work employing the Cone Calorimeter, glass-reinforced phenolics have been shown to have a lower rate of heat release than a FR glass-reinforced polyester over the entire test duration[11].

Phenolic composites have also been assessed in other fire tests, including the 2 foot tunnel test[12], oxygen index test[12–15], ASTM E–84 tunnel test[12], BS 476.6 fire propagation test[14,15], BS 476.7 surface spread of flame test[14,15], the NFP92–501 surface spread of flame test[14], the DIN 4102 surface spread of flame test[14], the NEN 3883 surface spread of flame test[14], the UL 94 vertical burn test[14], the OSU calorimeter[14], and the ASTM E-162 flame spread test[14].

3.2 Fire tests used

The above discussion indicates a paucity of information on the most suitable test procedures for assessing the fire performance of phenolic resins and composites, particularly where discrimination between formulations of similar behaviour is required. As indicated in the introduction, the tests were chosen on the basis of their use, both in Australia and overseas, and ability to assess materials of superior fire performance. It is also desirable that the tests provide a means of predicting the performance of the composites in their end-use situations. The following discussion of the tests selected encompasses the above selection criteria.

The first test considered in the project is the Australian Radiant Panel Test, AS 1530.3[1], the fire test currently used in Australia to control the use of building materials. It was developed in the 1950s[17] for wall linings, but is now used to control the flame propagation and smoke release properties of all materials used in nearly all classes of buildings, the exceptions being detached residential buildings and outbuildings. It relates a rate of increase in emitted radiation from the burning specimen to a predicted rate of flame spread up a wall corner, and takes the time at which flames are steady on the ceiling as a critical time in the growth of the fire[18], though others have disputed this[19]. Measurements are also made of ignitability, heat release and smoke release.

The next test chosen was the ASTM E-662[2] Smoke Chamber. This procedure has been one of the tests most commonly used to assess the smoke production of materials, and has previously been used in the assessment of glass-reinforced phenolic composites[12,13].

The ISO Ignitability Test (ISO 5657[3]) has recently been adopted in Australia (AS 1530.5[4]) as a test for assessing building materials, though it is not called up by Australian building regulations at this stage.

The fourth test chosen, the Cone Calorimeter, is the most sophisticated of the bench-scale tests selected, and is rapidly becoming one of the most common fire tests in the world. Not only has the test method been standardised by ASTM (ASTM E-1354[5]), but also by ISO and NFPA, and a joint Australian/New Zealand standard is being prepared. Its use is not restricted to building materials, and product standards are the normal avenue for providing specific requirements for particular products. At this stage it has no formal standing in Australia.

In addition to the bench-scale tests, full room burns were performed. After preliminary tests showed that flashover could not be attained with glass-reinforced phenolic composites using the ASTM burner programs[6], the more rigorous ISO burner program[7] was employed. Using this program might also allow an empirical relationship[20] between Cone Calorimeter data and flashover in the room to be evaluated.

3.3 Test attributes

The tests selected measure a variety of fire behaviour parameters. The Australian radiant panel test measures ignitability, flame propagation, heat release and smoke release. Ignitability is expressed as time to ignition, under the stepwise increases in radiation which are a feature of the test. Flame propagation is determined indirectly by measuring an increase in radiation as discussed above. For heat release, the test only measures radiant heat produced by the burning specimen, and the result normally reported is the total radiant heat released over a two-minute period following ignition. Smoke release is measured optically in the exhaust flue above the burning specimen. Although smoke is recorded continuously, only the maximum value of the average optical density over any one-minute period during the test, either before or after ignition, is normally reported.

The ASTM E-662[2] Smoke Chamber is designed solely to measure smoke production under both flaming and non-flaming conditions. Specimens are burnt inside a sealed chamber, and the smoke allowed to accumulate. Smoke is measured by an optical system registering a decrease in transmission, and the results reported as maximum specific optical density.

The ISO 5657[3] Ignitability Test measures time to ignition under selectable fixed radiation regimes. Results are reported as time to ignition under various irradiances, or the minimum irradiance needed to achieve ignition in a particular time.

In ASTM E-1354[4], the Cone Calorimeter method, measurements made include time to ignition, mass loss, heat release and effective heat of combustion. In addition, production of carbon monoxide and carbon dioxide are often measured, whilst there is also provision for soot mass sampling. Oxygen depletion is also monitored, though primarily for determining heat release. Ignitability is expressed as the time to ignition under selectable fixed radiation regimes. The smoke is measured optically in the exhaust flue. It is generally related to specimen surface area and mass loss, producing a parameter termed 'specific extinction area'. As the volume flow through the exhaust is measured, the total smoke produced can also be determined. The heat release measured is the total heat release, as it is determined from oxygen consumption.

The bench-scale tests use differing test conditions and specimen sizes and represent different fire scenarios. Thus there is not necessarily any correlation between the results of any two of them. Even the ISO 5657 Ignitability Test and the ASTM E-1354 Cone Calorimeter, which appear to measure ignitability under similar conditions, and have similarities in apparatus and technique, produce different results. Mikkola[(21)] has found that in many cases ignition times in the Cone Calorimeter are slightly higher than in the ISO Ignitability Test. In addition, for materials with short ignition times, the four-second cycle of the pilot flame in the ISO Ignitability Test (compared with constant application of the pilot in the Cone Calorimeter Test) becomes a source of significant differences between the two methods.

Specimen size varies greatly among the bench-scale tests (Table 3), with the AS 1530.3 Australian Radiant Panel requiring 46 times as much material for one test as the ASTM E-662 Smoke Chamber requires, or 18 times as much material as the ASTM E-1354 Cone Calorimeter requires (for assessment at three irradiance levels). The ISO 5657 Ignitability Test requires 4.5 times as much material as the ASTM E-1354 Cone Calorimeter to determine the ignition time at each irradiance level. These differences in material usage assume a major importance in the developmental stage of new materials, and unless the extra material used is offset by the quality and utility of the results obtained, it is very difficult to justify the use of material-expensive tests. In this project, it was not possible to make hand-batched specimens large enough for the AS 1530.3 Australian Radiant Panel Test, and production of sufficient 'tiles' to build up the necessary specimens was impractical in terms of time and material. Even hand-batched specimens for the ISO 5657 Ignitability Test were considered impractical when similar comparative data could be supplied by the Cone Calorimeter.

Table 3. Specimen replicates and sizes

Test method	Replicates required[a]	Size (mm)
(a) Bench-scale tests		
AS 1530.3 Australian Radiant Panel	6	600 x 450
ASTM E-662 Smoke Chamber	6 (3 per exposure)	76 x 76
ISO 5657 Ignitability	5 per irradiance level	165 x 165
ASTM E-1354 Cone Calorimeter	3 per irradiance level	100 x 100
Test method	Sheets required	Size (mm)
(b) Large-scale tests		
ASTM Room Fire Tests	8	1220 x 2440
[a]All methods require additional specimens if results vary too much.		

3.4 Behaviour of the phenolic specimens in the test procedures

The phenolic specimens showed a tendency to spall explosively in all tests (Table 4). The unreinforced phenolic (3U) disintegrated substantially when assessed in the ASTM E-1354 Cone Calorimeter, forcing the tests to be aborted. The reinforced phenolics (3R and 3F), when assessed in the ASTM E-662 Smoke Chamber using the flaming mode, also spalled to the point of forcing the tests to be aborted. The extent of spalling, for these specimens, appeared to have some dependence both on the irradiance and the presence of a flame (for ignition), though this relationship was not quantifiable. Spalling occurred more in the early stages of a test, and so in tests where ignition did not occur at the very start of the test (all except the ASTM E-662 Smoke Chamber Test – flaming mode), spalling had decreased or stopped before ignition occurred. In the case of the unreinforced phenolic (3U) in the ASTM E-1354 Cone Calorimeter, there was no material left after the initial spalling, and so ignition could not occur. The effect of the explosive spalling on test results was less marked in the ISO 5657 Ignitability Test and the AS 1530.3 Australian Radiant Panel Test; in both cases because explosive spalling had generally dropped to insignificant levels in the early stage of the tests.

Table 4. Occurrence of explosive spalling in bench-scale tests

Test method and exposure	Irradiance (kW/m^2)	Phenolic 3U	Phenolic 3R	Phenolic 3F
AS 1530.3 Australian Radiant Panel	2→25[a]			Some
ASTM E-662 Smoke Chamber:				
Non-flaming	25		Some	Some
Flaming	25		Excessive[b]	Excessive[b]
ISO 5657 Ignitability	30			Some
	40			Some
	50			Some
ASTM E-1354 Cone Calorimeter	35		Some	Some
	50	Excessive[b]	Some	Some
	75		Some	Some

[a] Estimated(4); radiation increases as test progresses.
[b] No result obtainable.

The comparatively benign fire test regime imposed by the AS 1530.3 Australian Radiant Panel test did not cause ignition of the phenolic composite (3F) tested in five out of six cases (Table 5). The level of combustion following the one instance of ignition was very low, as evidenced by the 'no result' for Flame Spread and the low Heat Evolved. Smoke Developed was low, though slightly higher for the case in which ignition occurred than for the other five tests where ignition did not occur.

Table 5. Ignitability, flame spread, heat evolved and smoke developed in AS 1530.3 Australia Radiant Panel test

Specimen	Ignitability		Flame spread		Heat evolved		Smoke developed	
	Time to ignition (min)	Index[a]	Flame spread time (min)	Index[a]	Heat evolved integral (kJ/m^2)	Index[a]	Maximum optical density (m^{-1})	Index[a]
Phenolic 3U	–	–	–	–	–	–	–	–
Phenolic 3R	–	–	–	–	–	–	–	–
Phenolic 3F	N[b] (16.7)	0 (3)	– (NF)	0 (0)	– (46.8)	0 (1)	0.020±0.005 (0.049)	2 (3)

Results are expressed as mean ± standard deviation of six replicates, unless otherwise specified. N indicates no ignition, and consequently no Flame Spread or Heat Evolved results. NF indicates no flame spread following ignition.
[a] Indexes for Ignitability are 0–20, and for the other parameters 0–10; low scores are better.
[b] Results for five replicates, as only one replicate ignited; results for the replicate that ignited are given in parentheses.

The ASTM E-662 Smoke Chamber has two modes for assessment – flaming and non-flaming. These phenolic composites were found to be unsuitable for assessment in the flaming mode due to explosive spalling (Table 6). In the non-flaming mode they achieved maximum specific optical densities at the low end of the scale, an order of magnitude lower than what have been obtained for some other composite materials[11].

Table 6. Maximum specific optical density in the ASTM E-662 smoke chamber

Specimen	Flaming	Non-flaming				
	D_m	$D_{m(corr)}$	Time to D_m (min)	D_m	$D_{m(corr)}$	Time to D_m (min)
Phenolic 3U	–	–	–	–	–	–
Phenolic 3R	N_{ex}	N_{ex}	N_{ex}	32±3	30±4	18±2
Phenolic 3F	N_{ex}	N_{ex}	N_{ex}	20±4	19±4	18±2

Results are expressed as mean ± standard deviation of three replicates.
N_{ex} indicates no result due to explosive spalling.

In the ISO 5657 Ignitability test, the explosive spalling which occurred prior to ignition is not believed to have affected ignition times (Table 7). Mikkola[21] has found differences in ignition time between the ISO 5657 Ignitability test and the ASTM E-1354 Cone Calorimeter. Whilst the test had a suitable range of conditions, in this project there was insufficient difference in the results (Figure 1) to justify the extra material required for the ISO 5657 Ignitability Test.

Table 7. Ignition times in the ISO 5657 Ignitability test

Specimen	Irradiance level (kW/m^2)	Time to ignition (s)
Phenolic 3U	–	–
Phenolic 3R	–	–
Phenolic 3F	30	N
	40	408
	50	255

Results are expressed as median of five replicates. N indicates no ignition.

The ASTM E-1354 Cone Calorimeter provides the most data of all the bench-scale tests assessed and, with the exception of the ASTM E-662 Smoke Chamber, uses less material than the other tests. Table 8 presents data on ignitability, heat release rate, heat of combustion and production of smoke and the gases carbon monoxide and carbon dioxide. Figures 2 and 3 show additional data, on mass loss rate and heat release rate versus time.

Table 8. Ignition time, rate of heat release, effective heat of combustion, and production of smoke and gases in the ASTM E-1354 Cone Calorimeter

Specimen	Irradiance level (kW/m^2)	Ignition time[a] (s)	Heat release rate (kW/m^2)		Average EHC[a] (MJ/kg)	Average SEA[a] (m^2/kg)	Average CO yield[a] (kg/kg)	Average CO_2 yield[a] (kg/kg)
			Peak	300 s ave.[b]				
Phenolic 3U	35	–	–	–	–	–	–	–
	50	N_{ex}	N_{ex}	N_{ex}	N_{ex}	N_{ex}	N_{ex}	N_{ex}
	75	–	–	–	–	–	–	–
Phenolic 3R	35	N	9±2	1.3±0.1	1.5±0.5	74±62	0.021±0.001	0.07±0.06
	50	206±25	88±1	62±4	11.5±0.7	160±29	0.010±0.001	0.75±0.05
	75	75±3	113±7	71±4	12.0±0.3	217±46	0.011±0.001	0.77±0.03
Phenolic 3F	35	529±19	68±17	51±12	9.6±4.6	117± 6	0.038±0.005	0.41±0.18
	50	226± 11	89±6	66±4	11.3±0.3	149± 10	0.017±0.001	0.68±0.03
	75	94±25	125±2	77±3	12.1±0.6	224±37	0.014±0.001	0.76±0.06

Results are expressed as mean ± standard deviation for three replicates. Horizontal orientation. N indicates no ignition. N_{ex} indicates no result due to explosive spalling. Some explosive spalling occurred in all tests, although largely prior to ignition.
[a] From start of test.
[b] From ignition.

The ASTM room fire tests, which were conducted using only one phenolic, did not result in flashover (Table 9). The maximum rate of heat released from the burning of the phenolic composite (Figure 4) was only about 100 kW.

Table 9. Event times in the ASTM Room Fire tests for Phenolic 3F

Event	Time (s)
Ignition of walls:	
• left	820 ± 160
• right	820 ± 160
Flashover criteria:	
• heat flux at floor 20 kW/m^2	
radiometer #1	N
radiometer #2	N
• paper targets ignite	
rear	800[a]
front	N
• flames out door	N
• temperature in upper layer 600°C	
room centre	635[a]
doorway	N
• heat release 1 MW[b]	N
Results are expressed as mean ± standard deviation for three replicates, unless otherwise specified. N indicates event did not occur.	
[a] Achieved in only one of the three room fire tests.	
[b] Flashover criterion in EUREFIC program.	

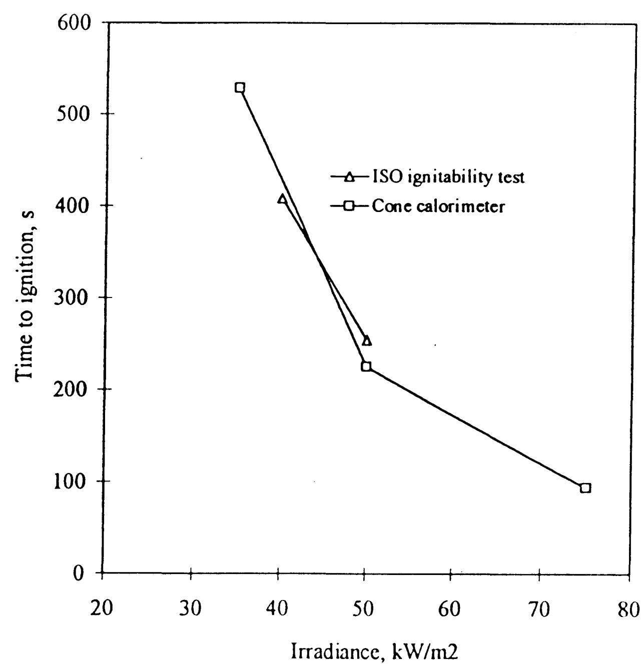

Figure 1. Ignition times in the ISO 5657 Ignitability Test and ASTM E-1354 Cone Calorimeter for Phenolic 3F.

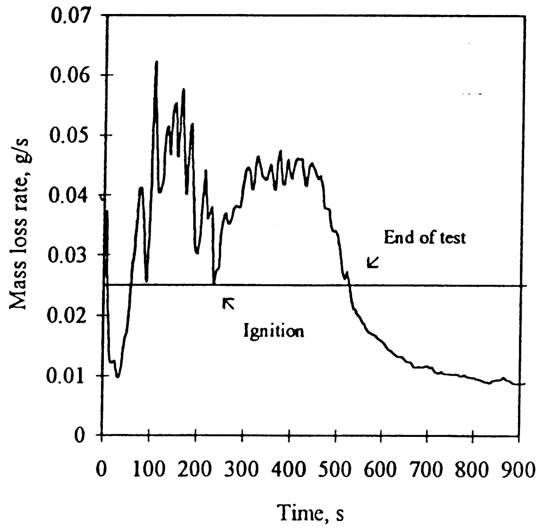

Figure 2. Mass loss rate of Phenolic 3F in ASTM E-1354 Cone Calorimeter showing spikes due to explosive spalling.

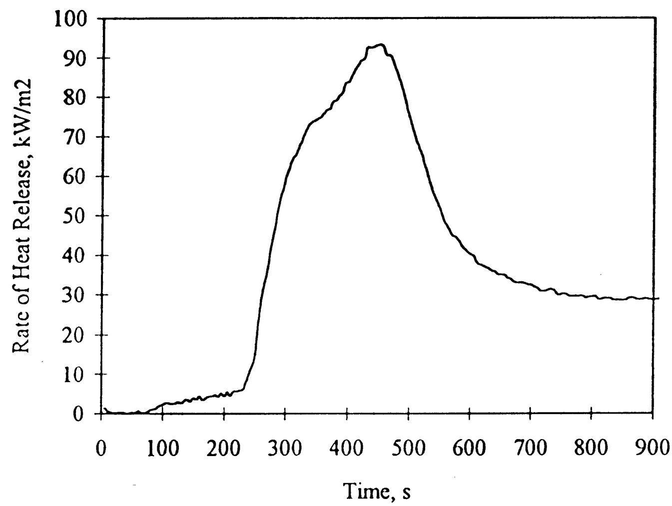

Figure 3. Rate of heat release of Phenolic 3F in the ASTM E-1354 Cone Calorimeter.

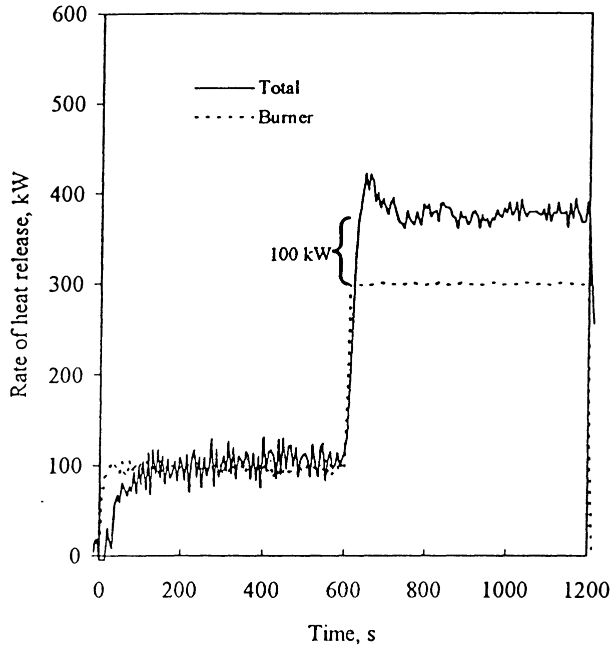

Figure 4. Rate of heat release of Phenolic 3F in the ASTM Room Fire Test.

Östmann and Tsantaridis[20] have developed an empirical relationship between ignition time and heat release in the Cone Calorimeter and time to flashover (defined as the time when a heat release of 1MW occurs) in room fire tests conducted to ISO 9705[7]. It had been hoped to compare results from this data with that prediction. However, Östmann and Tsantaridis'[20] relationship compared Cone Calorimeter data with room fire tests in which both walls and ceilings were lined with the test material, whereas in this project only the walls were lined with the test material, in accordance with the ASTM procedure[6]. Nevertheless, it was felt that it might be instructive to determine what the prediction was for the case where both walls and ceiling were lined with the test material, given that flashover did not occur with just the walls lined.

Östmann and Tsantaridis'[20] relationship is:

$$t_{fo} = 0.07 \frac{(t_{ig})^{0.25} \rho^{1.7}}{(THR_{300})^{1.3}} + 60$$

where t_{fo} = time to flashover in the ISO 9705 Room Fire test (sec);
t_{ig} = time to ignition in the Cone Calorimeter at 50 kW/m^2 (sec);
THR_{300} = total heat release during 300 seconds after ignition at 50 kW/m^2 in the Cone Calorimeter (mJ/m^2) ; and
ρ = specimen density (kg/m^3).

Using data from Tables 1 and 8 (THR_{300} = 300 x 300 sec Ave. Heat Release rate) gives t_{fo} = 1090 sec. Thus the prediction is that if walls and ceilings had been lined with the phenolic composite, flashover would have occurred at about 18 minutes in the 20-minute test.

4. CONCLUSIONS

The ASTM E-1354 Cone Calorimeter was found to be the most successful of the bench-scale tests for use in material development, even though it was not suitable for the unreinforced resin studied. The method is not yet cited in building regulations and thus it cannot be used for appraisal purposes. The range and severity of conditions available in the Cone Calorimeter method meant that it could be used successfully in comparing materials like the phenolic composites, which have favourable fire behaviour. Though explosive spalling did occur in the ASTM E-1354 Cone Calorimeter test, it was not as big a problem as it was in the ASTM E-662 Smoke Chamber when assessing phenolic composites.

The ASTM Room Fire tests of phenolic composite wall linings, using the ISO 9705 burner program, provided useful data, though flashover did not occur. Using Cone Calorimeter data, it was predicted that had wall and ceilings been lined with the phenolic composites, flashover would have occurred.

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