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THE PRODUCTION OF OXIDES OF CARBON FROM
THE THERMAL AND THERMAL-OXIDATIVE DECOMPOSITION
OF FLEXIBLE POLYURETHANE FOAMS

by

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POLYURETHANE FOAMS

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SUMMARY

The production of oxides of carbon from the thermal and thermal-oxidative decomposition of a polyester and polyether flexible polyurethane foam, parent polyols and yellow smoke has been studied at temperatures between 200 and 1000°C by gas chromatography. In inert atmospheres virtually the entire oxygen content of each foam is released as total oxides of carbon at 1000°C. At this temperature, the polyester foam releases 260 and 145 mg/g of carbon monoxide and carbon dioxide respectively whereas the polyether foam releases 400 and 42 mg/g respectively. Under oxidative conditions the maximum carbon monoxide yields (295 and 440 mg/g for the polyester and polyether foams respectively) are generated at 600°C whereas the maximum carbon dioxide yields are released at 1000°C (960 and 715 mg/g for the polyester and polyether materials).

KEY WORDS: Gas chromatography, mass spectrometry, polyurethane foam, pyrolysis,
toxic gas

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1. INTRODUCTION

Recent work in this laboratory¹ on the thermal decomposition of flexible polyurethane foams in air has been undertaken to monitor the release of nitrogen-containing materials, particularly hydrogen cyanide, under oxidative conditions. In this work hydrogen cyanide has been detected during isothermal decomposition experiments at temperatures above 600°C, reaches a 'peak' value at 700°C, decreases at 800°C, then increases again markedly towards 1000°C. These results coupled with other chromatographic evidence, have suggested that in this temperature region (600 to 1000°C) in air, there may be two distinct decomposition regions. Firstly at temperatures between 600 and 800°C there is a distinct oxidative region and secondly between 800 and 1000°C a region where non-oxidative pyrolysis is important. The reason for this is not understood but may be associated with the rapid release of volatiles during high temperature experiments which produces atmosphere vitiation near the sample in the furnace.

The work of this present report was undertaken to study the production of the carbon monoxide and carbon dioxide from the decomposition of certain flexible polyurethane foams and related materials. This information is required to understand the oxidation processes involved in the various temperature regions and is directly relevant to studies of toxic gas production in fires involving flexible polyurethane foams.

In this study the release of carbon monoxide and carbon dioxide has been monitored from the decomposition of polyester and polyether foams and respective parent polyols and from a sample of polyester yellow smoke^{2,3} (prepared under inert conditions). This work has been carried out both in inert and oxidative environments since the oxygen content of the foams, polyols and yellow smoke may be released as oxides of carbon during direct (ie non-oxidative) pyrolysis.

Material balances are presented to show the contributions of the oxygen and carbon contents of the foams to the production of oxides of carbon during the decomposition of the foams. Information about atmospheric vitiation gained from these balances is discussed.

2. EXPERIMENTAL

a) The Decomposition system

The decomposition experiments recorded in this report were carried out with an apparatus which has been widely used in this laboratory for studies of the decomposition products of various building and furnishing materials^{2,3,4}. In the apparatus, weighed samples of the foams, parent polyols and yellow smoke were decomposed in a small ceramic boat in a tube furnace (fitted with a silica furnace tube of approximately 18 mm I.D) in a flow of nitrogen (B.O.C. white-spot grade) or air (B.O.C. medical grade) for 15 minute intervals. The maximum temperature of the furnace is 1000°C with a temperature stability of better than ± 2 DegC at 1000°C.

The decomposition gases were collected over the total 15 minute decomposition in an evacuated plastic bag. The contents of the bag were thoroughly mixed before extracting samples for analysis.

b) Analysis of oxides of carbon

Samples of the gases from the plastic bag were removed with a syringe and analysed for carbon monoxide and carbon dioxide by gas chromatography. Details of this analysis have been recorded elsewhere⁵ but the analysis was modified to improve the sensitivity by replacing the silica gel column with Porapak Q (for carbon dioxide analysis) and increasing the 0.5 ml injection of gas onto the molecular sieve column (for the analysis of carbon monoxide) to 5 ml. This increase in sensitivity was required in order to limit the decompositions to the standard 10 mg* of foam (or equivalent weight of polyol and yellow smoke) which has been used throughout the work involving polyurethane foams. This is important in the case of polyurethane foams where atmosphere vitiation is suspected.

c) Materials

The polyester and polyether foams are identical to those used in earlier work^{1,2,3,6}. Both foams are based on T.D.I. The polyester foam is based on a lightly branched polyester of adipic acid and diethylene glycol and the polyether foam prepared from a polyether of glycerol, propylene oxide and ethylene oxide. Samples of the parent polyols were available.

*It has been the usual practice in the past to increase the sensitivity by decomposing larger quantities of sample.

The yellow smoke used in this work was prepared from the thermal (inert) decomposition of the polyester foam.

All decomposition experiments in this report used 10 mg of each foam or the equivalent weights of polyol and yellow smoke. It has been assumed that the yellow smoke, prepared from the polyester foam, is equivalent to that produced from the polyether material.

3. RESULTS

a) Decomposition of the foams, polyols and yellow smoke in inert atmospheres

The production of carbon monoxide and carbon dioxide from the decomposition of the polyester and polyether foams (10 mg) and equivalent weights of the parent polyols (7.3 and 7.5 mg for the polyester and polyether foam respectively) were studied at temperatures between 200 and 1000°C in a flow of oxygen-free nitrogen at 50 ml/min. As outlined in the experimental section the products were collected in an evacuated plastic bag (capacity 750 ml) and analysed by gas chromatography.

The yields of carbon monoxide and carbon dioxide from the polyester foam and equivalent weight of polyol are given in Fig.1. The data are recorded as yields per gram of foam. The data for the polyol therefore represent the contribution to the carbon monoxide and carbon dioxide yields from the polyol content of 1 gram of foam and the differences between the yields of the foam and polyol represents the contribution of the yellow smoke.

The data for the polyether foam and polyol is shown in Fig.2. For comparison purposes Fig.1 and 2 are recorded on the same scale.

It has been established in earlier work^{2,3} that the yellow smoke obtained from the polyester and polyether foams are essentially identical materials. Figure 3(a) shows the yields of carbon monoxide and carbon dioxide obtained from the decomposition of a polyester sample of yellow smoke (2.5 mg) and it will be assumed that this is also representative of the decomposition behaviour of polyether yellow smoke. The graph in Fig.3(a) is plotted as the yields per gram of foam in order to be directly comparable with the data of Fig.1 and 2; and is slightly approximated and assumes that the content of yellow smoke of each foam is 250 mg per gram of foam.

From Fig.1, 2 and 3(a) a number of points are apparent.

(i) Carbon dioxide is released in substantial quantities from the polyester foam at temperatures above about 300°C. This carbon dioxide is generated mainly from the polyol. Only very small quantities of carbon dioxide are generated from the yellow smoke.

(ii) Small quantities of carbon dioxide are generated from the polyether foam. The contribution from the polyol in this case appears to be small and inconsistent with the low yields observed from the polyester yellow smoke. The carbon monoxide released from the yellow smoke as obtained by the difference between the yields from the foam and polyol is somewhat larger than obtained experimentally in Fig.3(a).

(iii) Both foams release substantial quantities of carbon monoxide above 500°C. At temperatures up to 700°C this carbon monoxide is almost wholly derived from the polyol in each case. Between 700 and 1000°C the contribution of carbon monoxide from the yellow smoke is distinct in each case (Fig.1 and 2) and in good agreement with that expected from the decomposition of the yellow smoke alone (Fig.3(a)).

(iv) The temperature dependence of the carbon monoxide yields at high temperatures suggests that the yields might increase further at temperatures above 1000°C for both foams. This point and considerations of material balances during high temperature decomposition will be discussed further in subsequent sections of this report.

A summary of the yields of carbon monoxide and carbon dioxide from the foams, polyols and yellow smoke at 1000°C is given in Table 1.

Table 1

Maximum yields (1000°C) of carbon monoxide and carbon dioxide from the foams, polyols and yellow smoke in a nitrogen atmosphere. Units are milligrams per gram of foam in all cases.

Foam type	Product	Foam	Polyol	Yellow smoke
		(mg per gram of foam)		
Polyester	Carbon dioxide	145	156	5
Polyester	Carbon monoxide	260	198	58
Polyether	Carbon dioxide	42	5	-
Polyether	Carbon monoxide	404	304	-

b) Decomposition of the foams, polyols and yellow smoke in air

The decomposition experiments involving the foams, parent polyols and yellow smoke outlined above (Section 3A (a)) using oxygen-free nitrogen as the decomposition atmosphere were repeated using a flow of air.

The yields of carbon dioxide and carbon monoxide from the oxidative decomposition of the polyester foam (10 mg) and an equivalent weight of polyol (7.3 mg) are recorded in Fig.4. As before the yields are recorded per gram of foam.

The equivalent data for the release of carbon dioxide and carbon monoxide from the polyether foam (10 mg) and polyol (7.5 mg) are shown in Fig.5 and 6 respectively and the data for the polyester yellow smoke given in Fig.3(b). Again, it will be assumed that the behaviour of the polyester smoke is representative of the behaviour of the polyether smoke.

With reference to Fig.4, 5, 6 and 3(b) and the equivalent inert data of Fig.1, 2 and 3(a) the following observations can be made.

(i) The presence of oxygen markedly increases the yields of carbon dioxide throughout the whole temperature range (200 to 1000°C) for both the polyester and polyether foams (Fig.4 and 5 respectively). The form of the temperature - dependent behaviour suggests that this carbon dioxide is generated mainly by the polyol in each case. The differences in the carbon dioxide yields between each foam and respective polyol do not appear to correlate with the carbon dioxide yield from the yellow smoke.

(ii) The carbon dioxide yields for both foams in air (Fig.4 and 5) increase markedly with temperature to a 'peak value' at 600°C for the polyester foam and 700°C for the polyether foam. Above these respective temperatures the yields decrease towards 800°C then increase up to 1000°C. This behaviour is also present with the polyols alone particularly with the polyether polyol but is less distinct with the polyester. An equivalent behaviour is not detected in the carbon dioxide yields from the yellow smoke alone (Fig.3(b)) but a distinct discontinuity in the yield is observed at 800°C.

(iii) At low temperatures (200-500°C) the presence of oxygen increases the yields of carbon monoxide from each foam (Fig.4 and 6). Again this carbon monoxide is derived from the polyol in each case and the polyols appear to liberate more carbon monoxide when decomposed alone than when bonded in the polyurethane foam. At 600°C the carbon monoxide yields in air from both foams are maximum values. At temperatures above 600°C there is a rapid decrease in the yield of carbon monoxide in each case to a temperature of 900°C and then a slight increase in each case at 1000°C. This general behaviour is reflected in the behaviour of the respective polyols. The differences in the carbon monoxide yields between the foams and respective polyols do not clearly show the behaviour of the yellow smoke alone (Fig.3(b)). The yellow smoke does however show a distinct maximum value of carbon monoxide at 700°C.

(iv) With the polyester foam, the maximum carbon monoxide value in air at 600°C coincides with the temperature for the peak carbon dioxide value (Fig.4). With the polyether foam (Fig.5 and 6) the carbon dioxide peak value occurs at a slightly higher temperature (700°C) than observed for the carbon monoxide maximum (600°C).

A summary of the maximum observed yields of carbon dioxide and carbon monoxide from the thermal decomposition of the polyester and polyether foams, polyols and yellow smoke in an air atmosphere are recorded in Table 2. Also given are the decomposition temperatures at which these yields were observed.

Table 2

Maximum observed yields (and associated temperatures) of carbon dioxide and carbon monoxide from the thermal-oxidative decomposition of polyester and polyether foams, polyols and yellow smoke.

Units are milligrams per gram of foam in all cases.

Material	Carbon dioxide		Carbon monoxide	
	Temperature (°C)	Maximum observed yield (mg/g)*	Temperature (°C)	Maximum observed yield (mg/g)*
Polyester foam	1000	960	600	295
Polyester polyol	1000	865	600	330
Polyether foam	1000	715	600	440
Polyether polyol	1000	575	500	420
Yellow smoke ^φ	1000	535	700	88

* milligrams per gram of foam

^φ polyester yellow smoke

c) Material balances for decompositions in nitrogen

Material balances were carried out on the polyester and polyether foams and, because of the importance of the yellow smoke intermediate, on the polyester sample of yellow smoke. The elemental compositions of these materials, re-recorded from earlier reports for convenience^{2,3} are given in Table 3.

Table 3

Elemental compositions of the polyester and polyether foams and polyester yellow smoke

Material	Elemental composition*			
	C%	H%	N%	O%
Polyester foam	59.2	6.9	4.3	29.6
Polyether foam	61.3	9.0	4.1	25.6
Yellow smoke (polyester)	64.3	5.9	17.5	12.3

*C, H and N directly; O by difference from 100%

The direct material recoveries as weight, carbon and oxygen recoveries from the thermal decomposition of the foams and yellow smoke are given in Table 4. The data represent maximum recorded values (ie at 1000°C). The recoveries of the yellow smoke represent direct values, namely recoveries per gram of yellow smoke and not per gram of foam as used hitherto in this report.

Table 4

Direct material recoveries from the thermal (inert) decomposition of polyester and polyether foams and yellow smoke at 1000°C

Product	Direct recovery	Carbon recovery		Oxygen recovery	
	mg/g	mg/g	% Th*	mg/g	% Th*
Polyester foam					
Carbon dioxide	145	39.5	6.7	105	35.5
Carbon monoxide	260	111	18.7	149	50.3
Total	405	151	25.4	254	85.8
Polyether foam					
Carbon dioxide	42	11.5	1.9	30.5	11.9
Carbon monoxide	404	173	28.2	231	90.2
Total	446	185	30.1	261.5	102
Yellow smoke (polyester)					
Carbon dioxide	20 ^φ	5.5	0.85	14.5	11.8
Carbon monoxide	232 ^φ	99.4	15.5	133	108
Total	252 ^φ	105	16.4	147.5	120

*Per cent of the theoretical

^φYields per gram of yellow smoke

From Table 4, it is clear that between 40 and 45 per cent of the weight of each foam is released as total oxides of carbon. With the polyester foam approximately 26 per cent of the weight is released as carbon monoxide and 40 per cent for the polyether foam. With the foams the production of oxides of carbon involves 25 to 30 per cent of the carbon content and virtually the entire oxygen content. In fact in the case of the polyether foam the oxides of carbon account for a weight-loss slightly greater than the theoretical maximum based on the oxygen content. This is probably due to an inaccuracy in the oxygen content of the foam which is obtained indirectly (by a difference method) in elemental analysis.

In Fig.1 and 2 (in section 3A(a)) the form of the temperature-dependence of the evolution of the oxides of carbon suggested that at temperatures in excess of 1000°C the yields might be substantially higher than observed at the maximum temperatures of 1000°C. Clearly the data of Table 4 shows that the results at 1000°C are oxygen-limited and experiments in excess of 1000°C would not substantially increase the yields.

With the yellow smoke (Table 4) approximately 25 per cent of the theoretical weight loss is released as oxides of carbon with 23 per cent being carbon monoxide. The total oxides of carbon account for approximately 16 per cent of the theoretical carbon content and 120 per cent of the oxygen content. The observed figure of 120 per cent is in this case in error in one of two ways. Firstly because of a possible inaccuracy in the oxygen content of the smoke because of the indirect method of analysis and secondly an error due to polyol contamination since the polyol contains nearly 40 per cent by weight of oxygen. An error because of contamination of this kind was anticipated before undertaking the experiments with the yellow smoke. The results of Table 4 are interpreted as suggesting that, like the foams, the entire oxygen content of the yellow smoke is released as oxides of carbon at 1000°C.

d) Material balances for decomposition in air

The material recoveries obtained during the thermal-oxidative decomposition of the foam and yellow smoke are recorded in Table 5. The data is presented as weight of carbon recovery both at 1000°C and at the temperature corresponding to maximum carbon monoxide release.

With the two foams, 56.1 and 56.9 per cent of the carbon content of the polyester and polyether foams respectively are released at 1000°C as oxides of carbon. At 600°C the total carbon recovery from the polyester foam is 60.6 per cent (somewhat greater than observed at 1000°C); with the polyether foam the carbon recovery is 45.5 per cent but is complicated by the fact that in this case the maximum carbon monoxide release occurs at a somewhat different temperature to the maximum carbon dioxide release. At 700°C the total carbon recovery is 52.5 per cent which is almost the same as observed at 1000°C.

This evidence supports the idea that, under the decomposition conditions used in this report, there is a two-region decomposition. Namely that at temperatures of around 600 to 700°C there is a distinct oxidative region whereas

at higher temperatures the rapid release of products coupled with rapid uptake of oxygen from the air stream by the sample may cause some atmospheric vitiation and a reversion to some pyrolytic decomposition behaviour. This problem may also be interrelated to other important phenomena such as the effects of oxygen and temperature on the degradation of the foams to produce large molecular species which are volatile at the high temperatures used in this work. For example the presence of oxygen may alter effective contact times of the foams within the furnace because of increasing the volatilization of the polyol and yellow smoke fragments. This will be discussed further in a later section.

Table 5

Material recoveries from the thermal oxidative decomposition of polyester and polyether foam and polyester yellow smoke at 1000°C and at the temperature of maximum carbon monoxide release

Material	Yields at 1000°C			Yields at maximum carbon monoxide			
	Direct recovery mg/g	Carbon recovery		Temp (°C)	Direct recovery mg/g	Carbon recovery	
		mg/g	% Th*			mg/g	% Th*
Polyester foam							
Carbon dioxide	960	262	44.2	600	850	232	39.2
Carbon monoxide	165	70.7	11.9	600	295	126	21.4
Total	1125	332.7	56.1		1145	358	60.6
Polyether foam							
Carbon dioxide	715	195	31.8	600	330	90.0	14.7
Carbon monoxide	360	154	25.1	600	440	189	30.8
Total	1075	349	56.9		770	279	45.5
Yellow smoke (polyester)							
Carbon dioxide	2140 ^φ	584	90.8	700	1000	273	42.4
Carbon monoxide	112 ^φ	48	7.5	700	342	147	22.8
Total	2252 ^φ	632	98.3		1342	420	65.2

*Per cent of the theoretical

^φYields per gram of yellow smoke

In air (Table 5) there is an almost complete recovery of the total carbon content of the yellow smoke at 1000°C. At 600°C the carbon recovery is 65.2%. The extent of oxidation of the yellow smoke is much greater when decomposed alone than when generated in situ from the foam (see Fig.3(b) 4, 5 and 6).

4. DISCUSSION

In the studies of the production of oxides of carbon during the thermal and thermal-oxidative decomposition of the polyester and polyether foams and polyols and the polyester yellow smoke, a number of important points have emerged.

Under inert conditions, carbon monoxide is an important decomposition product from both foams at temperatures above 500°C. At temperatures up to 800°C the carbon monoxide is, in each case, produced from the respective polyol. At temperatures above 800°C there is a distinct contribution from the yellow smoke.

Carbon dioxide is an important product from the polyester foam under inert conditions, but a minor product from the polyether material. At 1000°C, the highest temperature used in this work, almost the entire oxygen content of each foam is released as oxides of carbon.

In oxidative environments the yields of carbon monoxide increase with temperature to 600°C then decrease markedly in each case, showing a slight increase again between 900 and 1000°C. This general behaviour is also reflected in the yields of carbon dioxide. Material recovery calculations show that the total carbon recovered as oxides of carbon for the polyester foam at 600°C is greater than observed at 1000°C. With the polyether foam the carbon recovery at 600°C is only slightly less than observed at 1000°C. This has been explained by atmosphere vitiation in that during oxidative decomposition at temperatures above about 600°C the rate of release of volatiles from the foams is high enough to restrict the access of air to the material and coupled with the rapid uptake of oxygen by the sample produces a vitiated atmosphere near the sample. This effect, as mentioned before, may be complicated because of the probable alteration in contact times of these materials in the hot furnace as a result of oxygen fission producing an increase in the rate of degradation into large, but volatile, molecular species - e.g. fragmented polyol.

In general, the temperature dependence of the formation of oxides of carbon is in agreement with the results of earlier studies¹ of the nitrogen containing products from the thermal oxidative decomposition of the foams, polyols and yellow smoke. In this work the production of hydrogen cyanide (the main nitrogen

containing product) is detected in air atmospheres at temperatures of 600°C, reaches a 'peak' value at 700°C, decreases at 800°C then rises again at 900 and 1000°C.

In the light of the data from the present work, it now appears that the decomposition of the yellow smoke (whether decomposed directly or indirectly) to produce hydrogen cyanide is 'catalysed' by oxygen. Hydrogen cyanide (and certain other organic cyanides) are detected in air at temperatures of 600 and 700°C but at 800°C the rate of release of volatiles turned the mode of decomposition towards pyrolysis conditions resulting in a reduction in the yields of cyanide. At temperatures of 900 and 1000°C the cyanide yields rise due to the 'inert' temperature dependence.

5. CONCLUSIONS

1. Carbon monoxide is an important product during the inert decomposition of both the polyester and polyether foams at temperatures above about 500°C. At 1000°C approximately 260 and 404 mg of carbon monoxide are released from 1 gram of the polyester and polyether foams respectively.
2. 1 gram of the polyester foam releases 145 mg of carbon dioxide under inert conditions at 1000°C. The polyether foam under these conditions releases 42 mg/g.
3. At 1000°C virtually the entire oxygen content of each foam is released under the inert environment as oxides of carbon.
4. Under oxidative conditions, the maximum carbon monoxide yields are released at 600°C (295 and 440 mg/g for the polyester and polyether foams respectively).
5. Under oxidative conditions, the maximum carbon dioxide yields are recorded at 1000°C as 960 and 715 mg/g for the polyester and polyether foams respectively.
6. Material recoveries of carbon as total oxides of carbon under oxidative conditions show that the main oxidation region lies at temperatures between 600 and 700°C. At temperatures above this region, atmosphere vitiation (due to rapid product release and rapid consumption of oxygen by the sample) causes a reversion to some pyrolytic decomposition behaviour.

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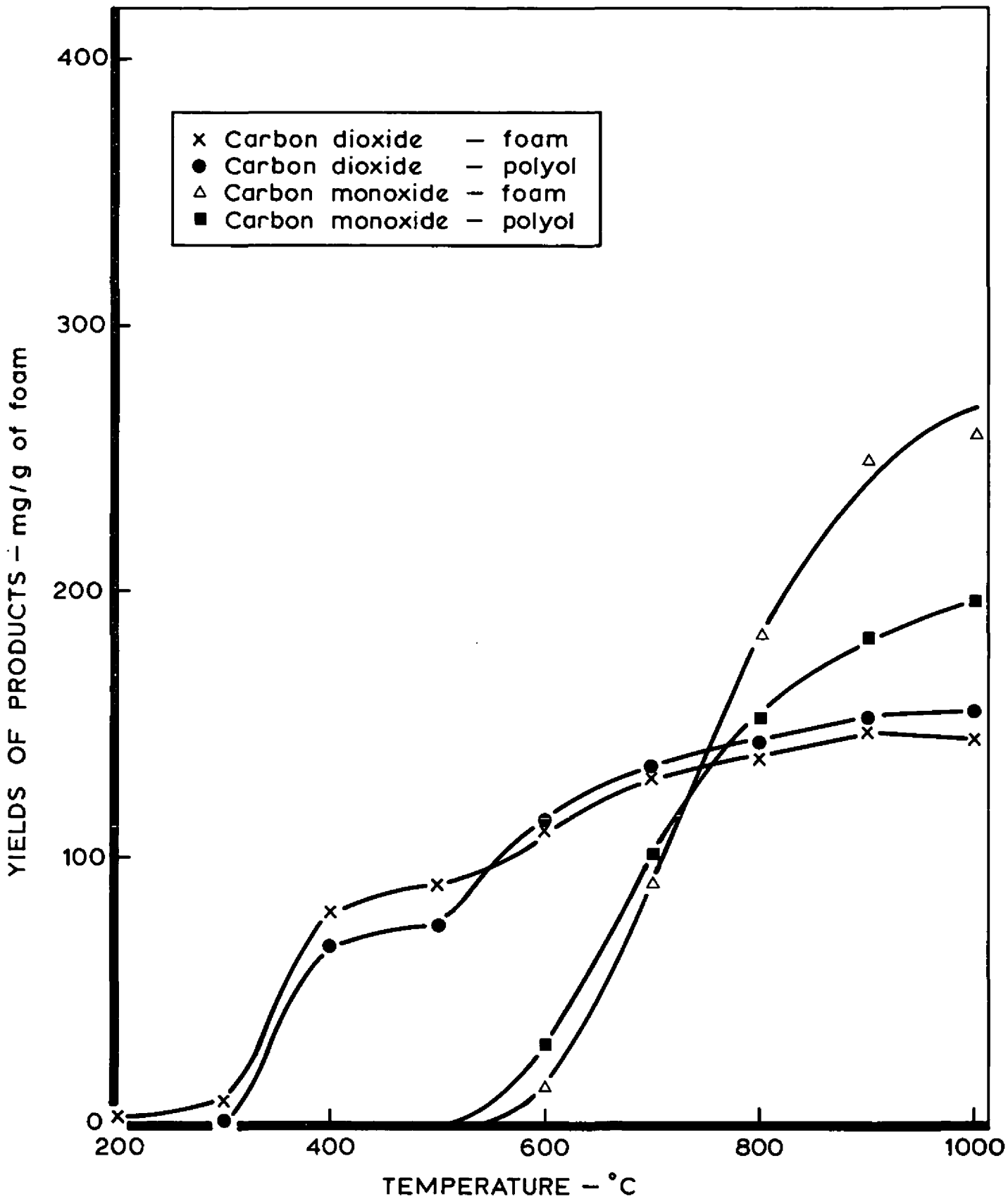


FIG 1 THE PRODUCTION OF CARBON DIOXIDE AND CARBON MONOXIDE FROM THE THERMAL (INERT) DECOMPOSITION OF POLYESTER FOAM AND POLYOL

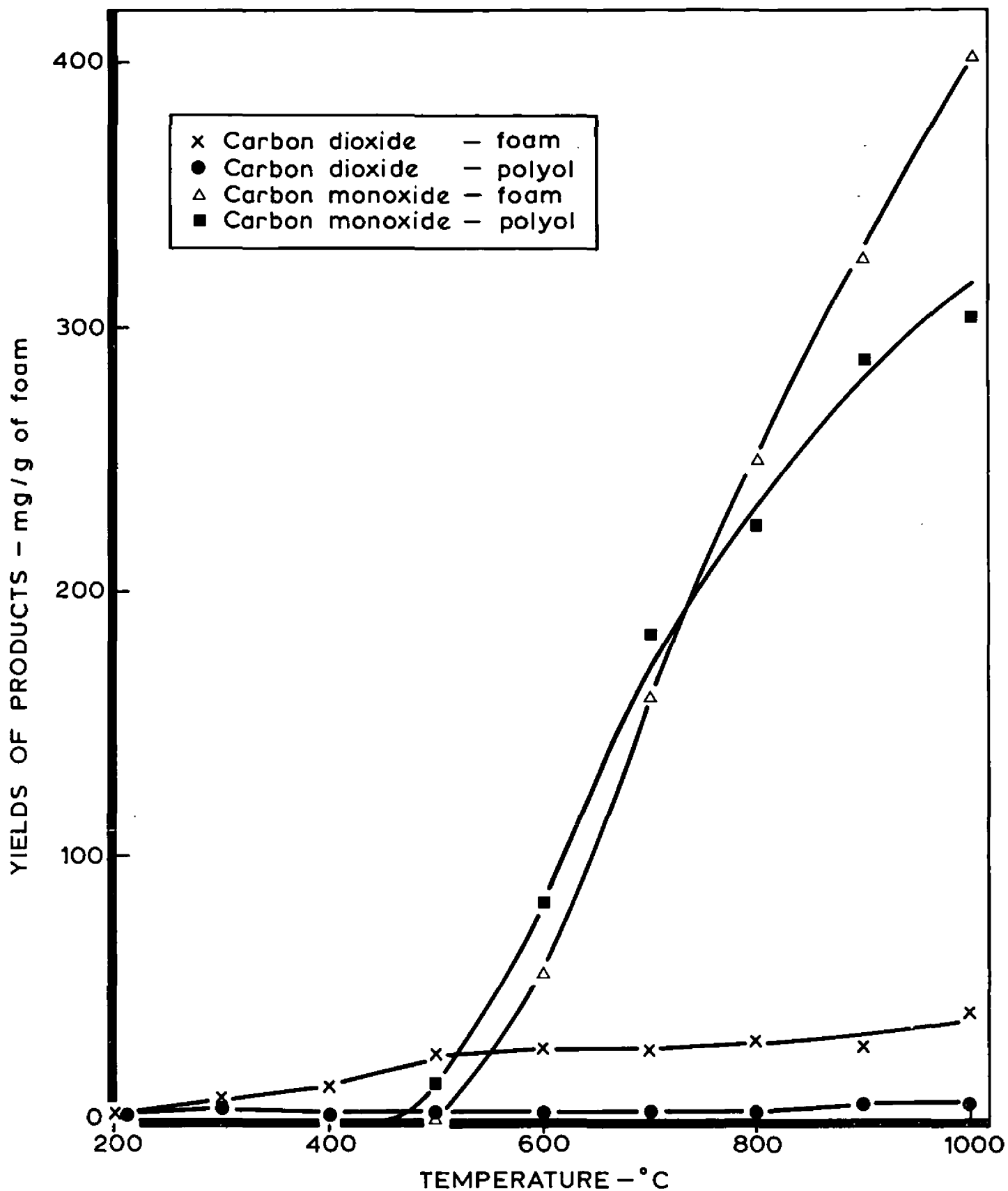


FIG 2 THE PRODUCTION OF CARBON DIOXIDE AND CARBON MONOXIDE FROM THE THERMAL (INERT) DECOMPOSITION OF POLYETHER FOAM AND POLYOL

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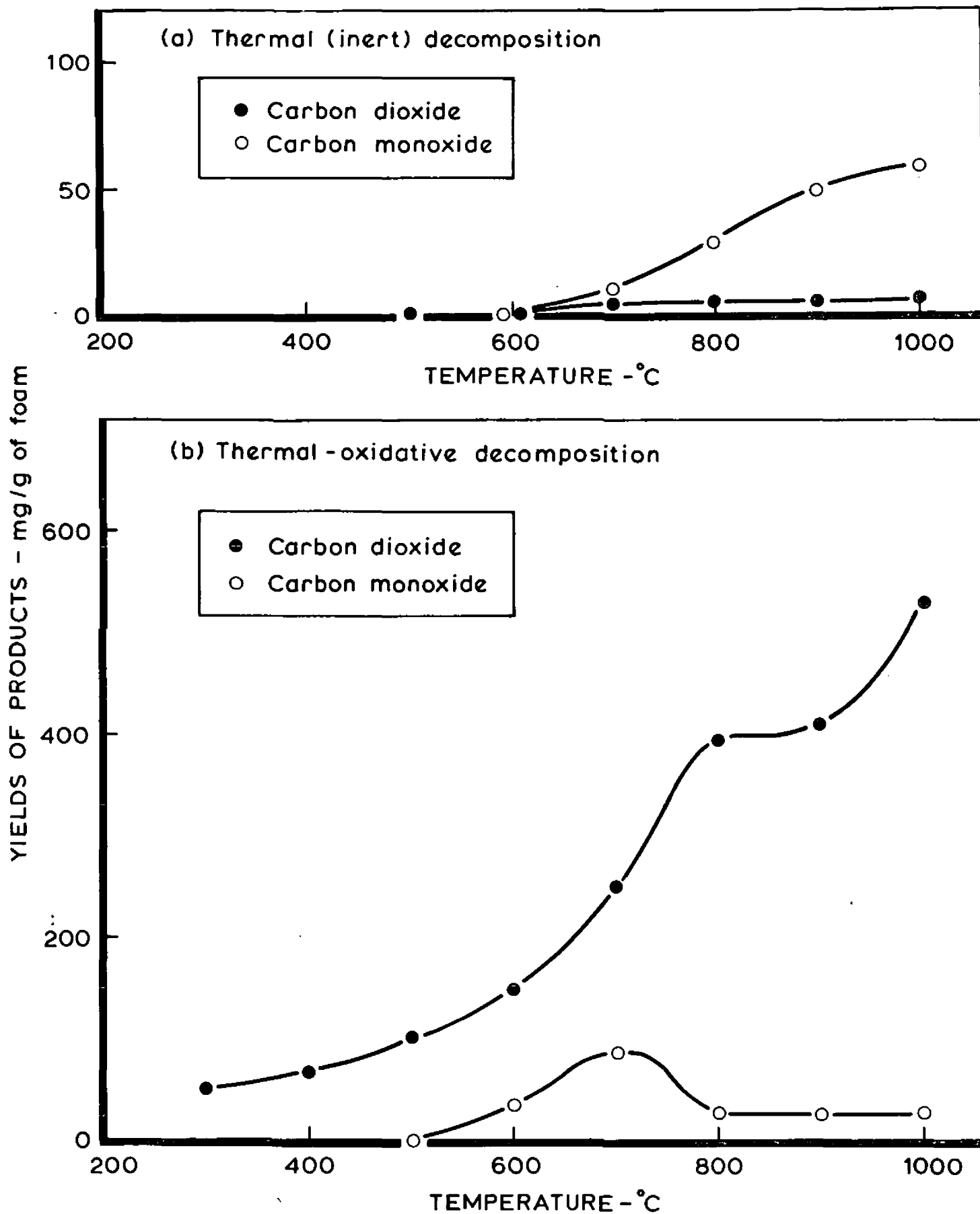


FIG 3(a) and (b) THE PRODUCTION OF CARBON DIOXIDE AND CARBON MONOXIDE FROM THE THERMAL AND THERMAL -OXIDATIVE DECOMPOSITION OF YELLOW SMOKE

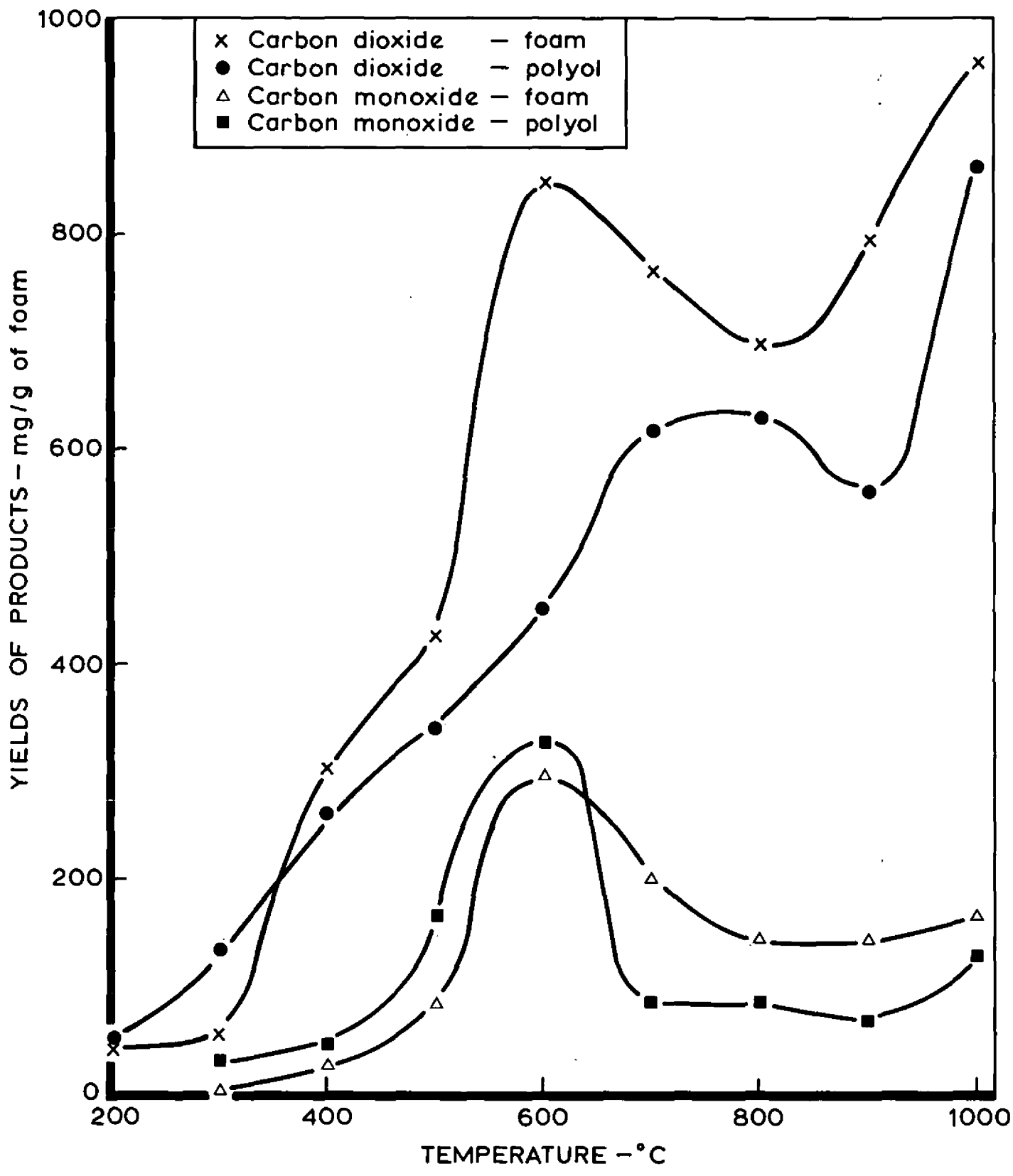


FIG 4 THE PRODUCTION OF CARBON DIOXIDE AND CARBON MONOXIDE FROM THE THERMAL- OXIDATIVE DECOMPOSITION OF POLYESTER FOAM AND POLYOL

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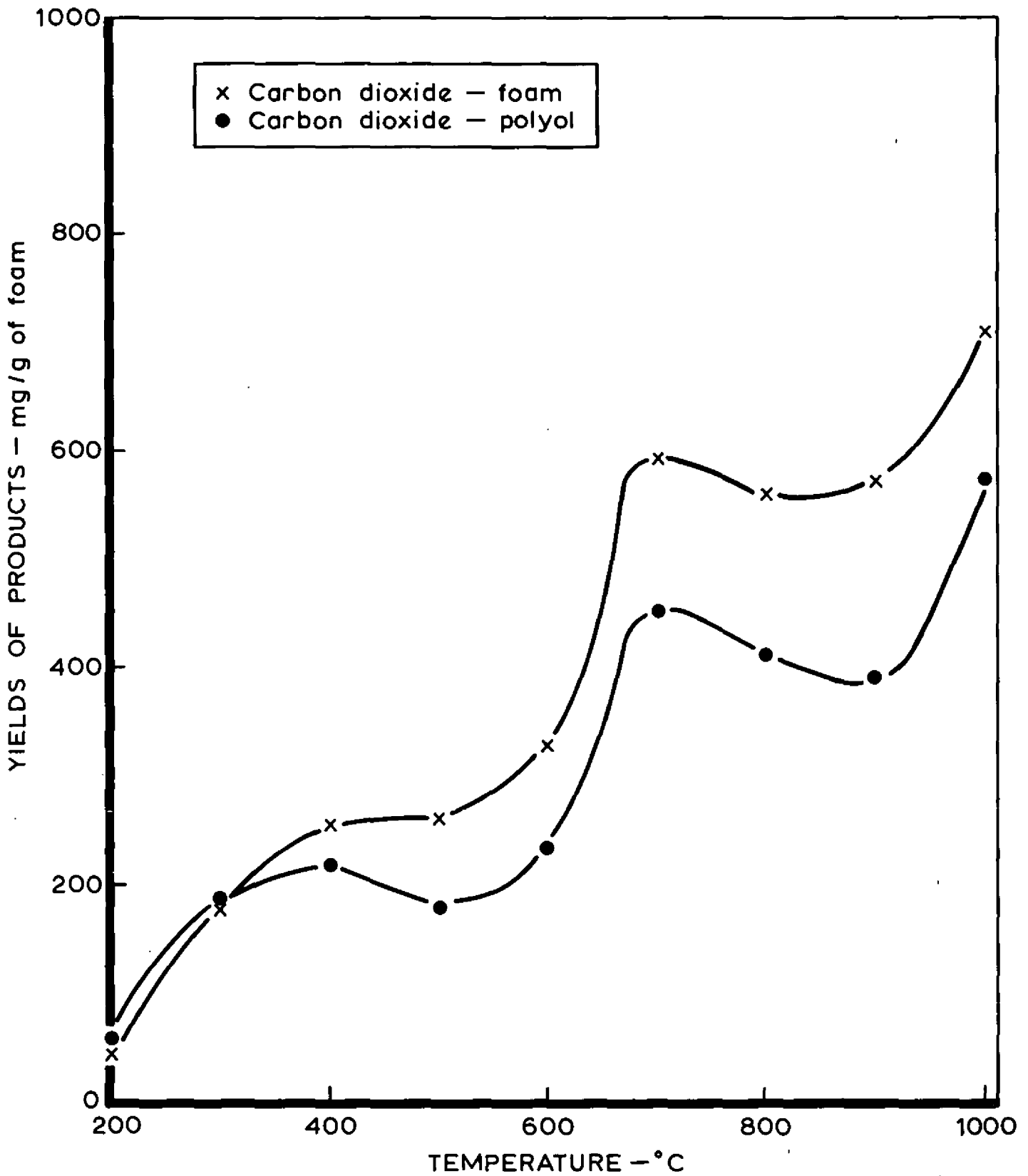


FIG 5 THE PRODUCTION OF CARBON DIOXIDE FROM THE THERMAL-OXIDATIVE DECOMPOSITION OF POLYETHER FOAM AND POLYOL

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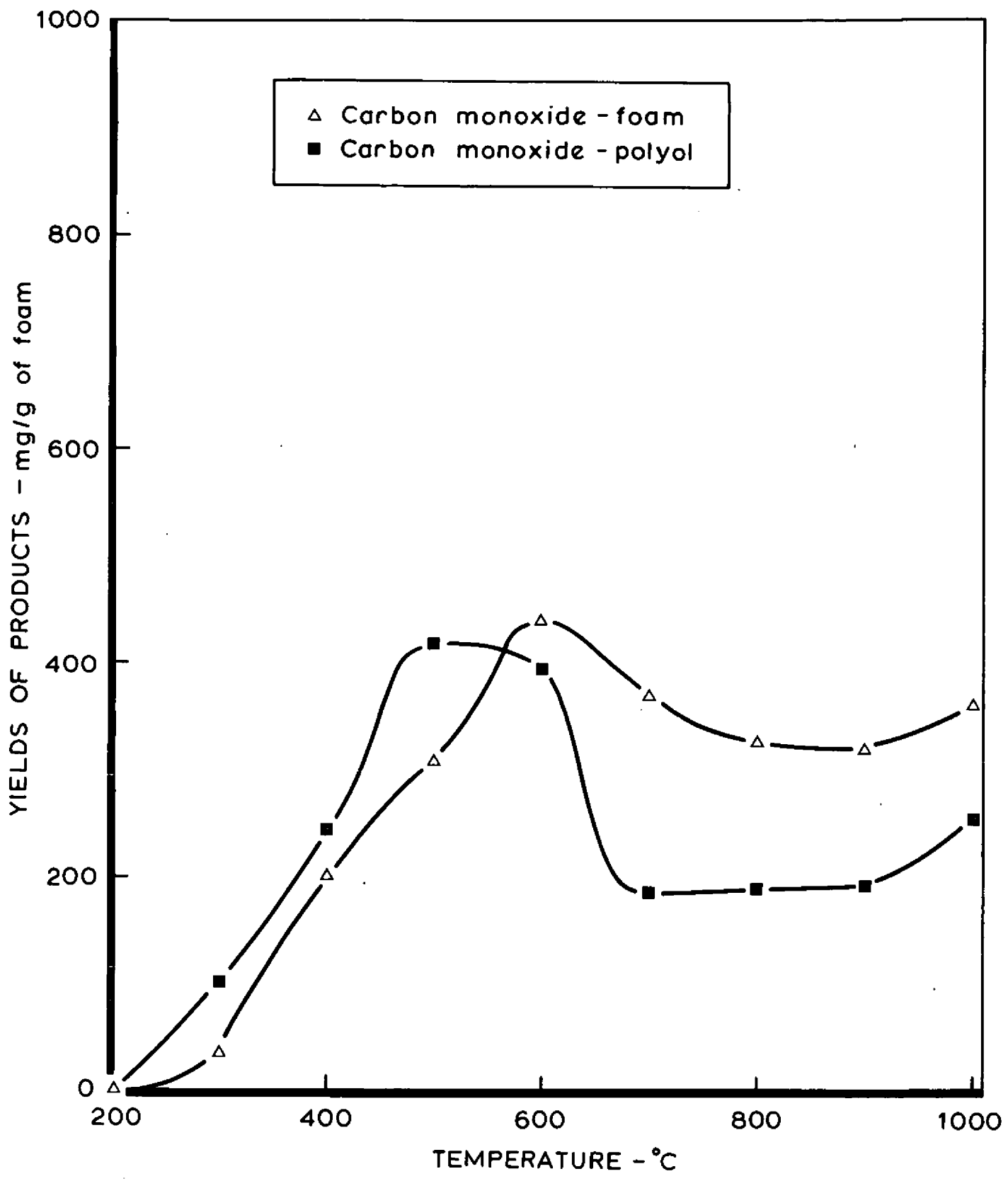


FIG 6 THE PRODUCTION OF CARBON MONOXIDE FROM THE THERMAL - OXIDATIVE DECOMPOSITION OF POLYETHER FOAM AND POLYOL

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