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CARBON ATTRITION DURING THE FLUIDIZED COMBUSTION OF COAL

Quarterly Technical Progress Report for the Period December 1, 1983—February 29, 1984

By
Leopoldo Massimilla
Riccardo Chirone
Matteo D'Amore

Work Performed Under Contract No. FG22-81PC40796

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Naples, Italy

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SUMMARY

Batch fluidized bed combustion of 5 mm particles of petroleum coke has been carried out in the 40 mm I.D. combustors previously described (DOE Reports No DOE/PC40796-1 and 2), to investigate the generation of fines due to the abrasion of bed solids on the surface of burning particles.

The modification produced in the texture of the cortical region of coke particles by combustion have been examined under an optical microscope and related to the "activation" of the surface in respect to attrition.

A descriptive model of combustion assisted attrition of particles of petroleum coke burning in a fluidized bed has been proposed.

INTRODUCTION

The elutriation of carbon fines is a drawback of the utilization of fluidized bed technique in coal combustion and gasification. Although carbon carryover has long been regarded as a problem in the design of efficient and environmentally acceptable fluidized coal combustors and gasifiers (1,2,3,4), only recently phenomena controlling the generation of carbon fines under conditions typical of fluidized combustion have been considered in detail. Beér et al. (5) pointed out the interaction between coal fragmentation due to devolatilization, char attrition by abrasion and reduction of particles of burning char to elutriable sizes in determining the flow rate of entrained carbon. Others found that a simple relationship applied between elutriation rates of attrited carbon and bed carbon loadings and particle size distributions when burning a bituminous coal in a continuously operated fluidized combustor. An empirical attrition rate constant of the coal tested was obtained as a result of the experiments (6,7). Using this constant, model calculations were performed to outline the attrition contribution to carbon elutriation in a fluidized coal combustor (5,8), and investigate the influence of fragmentation and related char size reduction on bed carbon loading and carbon loss by attrition (9).

The present work deals with the generation of fines during the fluidized combustion of petroleum coke. Such material, previously used by La Nauze and Jung (10,11) to investigate the rates of mass transfer of oxygen to particles burning in a fluidized bed, provides a carbon which is substantially free of volatiles and ashes. The interfering effects of devolatilization and fragmentation on attrition are, therefore, avoided and the texture of the particle which is relevant to attrition, essentially depends on the pattern of stratification of the combustible matter in the particle. Moreover, the combustion kinetics of a petroleum coke is known (12). A batch fluidized combustion technique has been used. It affords the time resolution of attrited fines generation as well as the study of the influence of combustion on attrition by changing oxygen concentration in the inlet fluidizing gas. This includes the extreme case in which the bed, kept at the combustion temperature by external heating, is fluidized by inert gas so that rates of elutriated carbon fines are representative of purely mechanical attrition without the influence of combustion.

EXPERIMENTAL

The chemical analysis and the physical properties of the petroleum coke were:

- Proximate analysis (weight % on dry basis)

Volatile matter = 0.44
Ash = 0.26
Fixed carbon = 99.3

- Physical properties:

Particle density = 1.64 g cm^{-3}
True density = 2.12 g cm^{-3}
Porosity = 22.6%
Internal surface area $\approx$ 1 m^2 g^{-1}

Batches of 21 approximately 5 mm particles, totalling always the same amount w_{ci} of carbon (2 g), were injected into the combustor in each run. The bed was made of 0.3-0.4 mm sand , the unexpanded bed height being about 100 mm. Bed temperature was 850°C, with a fluidizing velocity of 80 cm s^{-1} at this temperature. Air containing 21, 10, 4.5 and 1.4% O_2, and nitrogen, were used as inlet fluidizing gases. Oxygen concentrations at the outlet varied from 18.5, 8.8, 3.8 and 1.2%, immediately after charging the coke, to the corresponding inlet values at the burn-out. Measurements were made without and with pretreatment of coke in nitrogen fluidized beds.

A basket—equipped combustor and a two-exit head combustor, both 40 mm ID and 1 m high, were used. Details about design and operation of these combustors are given elsewhere (13). Briefly, with the basket-equipped combustor it was possible to collect particles and measure particle shrinkage, carbon content of the bed w_c and burn-off extent $\xi = (w_{ci} - w_c)/w_{ci}$ at any time during the run. The two-exit head combustor allowed the collection in separate filters of attrited carbon elutriated from the bed at various burn-offs. The accumulation in the filters of amounts of fines large enough to make weighing and chemical analysis reliable was obtained by cyclical runs involving various batches of coke for each experimental condition. Postcombustion of attrited fines was minimized by taking the combustor freeboard at low temperature. Separate tests carried out by feeding fines of the same size as those generated by attrition indicated that their average residence time in the bed and freeboard was about 1 min. This time was at least one order of magnitude smaller than the characteristic times of coke combustion and attrition. When using the two-exit head combustor the burn-off was monitored by means of gas analysis. Burn-out times, given in Table 1, were determined as the times at which CO_2 concentration in outlet gases dropped to zero.

Sizes of feed and partially burnt particles were measured by a micrometer in each of three perpendicular planes. Shape factors ϕ_1, defined by $D=\phi_1 d$ where D is the diameter of the sphere having the same mass as the particle and d is the average of the particle sizes, were calculated. The shape factor ϕ_{1i} of feed particles was 0.91. The contours of the cross sections of coke particles were examined under an optical microscope (Fig. 1A and B). The specimens were obtained by inbedding the particle in a cylinder of epoxide resins, cutting the cylinder across the particle and polishing the surface to be observed. Surface based shape factors ϕ_2 were estimated by measuring the lengths of the contours of cross sections of particles which had been submitted to nitrogen pretreatment. Again the relationship $D=\phi_2 d$ was used, but in this case D is the diameter of the sphere having the same external surface as the particle. Values of ϕ_2 were about 1.2. Attrited carbon fines collected at the filters were also observed under a microscope. Their size ranges and Sauter average diameters are given in Table 1.

RESULTS AND DISCUSSION

Burning model and particle shrinkage rates

The ratios N/N_i of the number of particles collected from the combustor to the original number of particles of the batch of coke are given in Fig. 2 as a function of the fractional time for complete conversion t/t_{bo} and extent of burn-off ξ . As expected there was no fragmentation due to devolatilization. $N/N_i<1$ found for $t/t_{bo}>0.6$ might be related to differences in burn-out times of particles of the same batch or, possibly, to the structural collapse of some particles at high ξ and faster burn-off of resulting subparticles.

Average diameters of particles collected at various times are compared in Fig. 3 with the relationship:

$$\frac{d}{d_i} = (1-\xi)^{1/3} \qquad (1)$$

valid for burning at constant particle density. The ratio ϕ_{1i}/ϕ_1 which should appear as factor of the second member of Eq. 1 (10) is supposed to be 1 for any d from d_i to 0. The comparison indicates that coke combustion essentially follows the shrinking particle model. Some internal burning, however, might take place, as noted by previous authors (10). Figure 1B suggests that this certainly occurred in the cortical region of the particle, producing changes in the original texture of coke relevant to attrition.

Calculations indicate that the fractional kinetic resistance to combustion was less than 5% for ξ up to 0.75 at least under all the experimental conditions tested. The Sherwood numbers ($Sh=k_g d\, \phi_1/D_g$) for the burning particles were determined using the experimental data with the assumption that carbon was converted into CO_2 close to the particle surface and that oxygen concentration in the bed was that measured at the combustor outlet. Values of Sh are given in Table 1 together with the particle diameter for which they were calculated. They are about one half smaller than those previously measured in experiments carried out with single coke particles (10,11) but, being ε_b the average bed voidage, double the value

$Sh=2\,\varepsilon_b$ suggested as the lower limit in the absence of any forced convection $^{(15)}$. This condition might have been to some extent approached with the relatively fine sand used in the present work and consequent low gas velocity in the bed particulate phase. Neglecting kinetics resistance, the ratios d/d_i experimentally determined as a function of the fractional time t/t_{bo} are compared in Fig. 4 with curves derived assuming film diffusion control according to the equation

$$\frac{d}{d_i} = (1-t/t_{bo})^a \qquad (2)$$

with a=2/3 or 1/2 in turbulent and Stokes regime respectively $^{(16)}$. Most data fall inside or on the boundaries of the area defined by the two relationships. Also in this case results were correlated with the assumption of constant particle shape factor throughout burn-off.

Attrition curves

Carbon attrition rates measured as a function of time using nitrogen and air as fluidizing gases are reported in Fig. 5. The curve with nitrogen is typical of purely mechanical attrition of angular solids in fluidized beds $^{(17)}$. The peak value of e_c at t=0 is the initial attrition rate with fresh coke surface. Rounding off irregularities results in the decay of elutriation rate to a low quasi-stationary value of e_c. Figure 1A shows the contour of particle surface in these conditions. A peak at t=0 is also found with air, but in this case, after a temporary decay, e_c increases again to reach a maximum e'_c at t=t'. From this, e_c eventually declines to become zero at the burn-out time. Similar attrition curves were obtained with 10, 4.5 and 1.4% O_2 in fluidizing gas.

The comparison between attrition curves for operation with nitrogen and with oxygen-nitrogen mixtures suggests that attrition is enhanced by combustion. This is confirmed by experiments presented in Fig. 6. After 60 min pretreatment with nitrogen the fluidizing gas was switched to air (Fig. 6A). The experiment was repeated (Fig. 6B), but in this case fluidizing gas was changed again from air to nitrogen as the top level of e_c was reached at t=t'. The rapid increase and decrease of e_c reflect the change from a purely mechanical attrition to a combustion assisted attrition, and viceversa, in both cases. Attrition data reported in Fig. 7 for different oxygen concentration in fluidizing gas were obtained with 60 min pretreatment with nitrogen. The abscissa t=0 marks the switching from nitrogen to oxygen containing gas. The overall combustion efficiencies $\eta=(w_{ci}-\int_0^{t_{bo}} e_c dt)/w_{ci}$ found in these runs are reported in Table 1. Instantaneous η evaluated at t=t' are also given in the table.

Analysis of attrition data, together with information provided by microphotographs of Fig. 1A and B, suggests that burning speeds up fines generation by a process of "activation" of the surface. With this view, the trends of e_c in Fig. 7 can be seen as the result of two opposite effects of burn-off: the uneven progress of the combustion front which leads to the generation and growing of detachable asperities and the reduction of the total number of detachable asperities because of particle shrinking. If so, specific attrition rates $e_c/N \pi d^2 \phi_2^2$ obtained combining data in Figs. 2,4 and 7, should show, as a function of t/t_{bo}, the approach to and the establishment of a steady state condition with the surface of coke particles fully activated in respect to attrition. This expectation is in good, although not total agreement with experimental findings in Fig. 8. For all the series of runs, specific attrition rates tend to increase rapidly somewhere after reaching, or even only approaching, a plateau. The deviation from the steady state regime of surface activation, particularly relevant when operating with 21 and 10% O_2, may be associated with internal burning and fragilization of coke particles at high burn-off.

A descriptive model of combustion assisted attrition

The model is based on the assumption that channels and macropores appearing in petroleum coke (Fig. 1A) divide the particle into elements burning at different rates because of their different accessibility to oxygen. Elements reacting more slowly emerge as asperities on the external surface of the particle (Fig. 1B and C). They eventually burn or are abraded by bed solids, but are regenerated along the same or other directions by the uneven progress of the combustion front. Attrition is a discontinuous phenomenon, which intermittently involves elements in their entirity. Focusing on attrition, the model further assumes that combustion also leads to the periodic disappearance of whole elements. Their size is equal to that of the elements forming asperities, which in turn is equal to that of attrited fines. The rate of removal of elements by combustion can be evaluated by accounting for the rate of diffusion of oxygen from the gas phase to the particle; that of removal by attrition depends on bed agitation and mechanical resistance of asperities.

Considering elements of roughly cubic shape, the relationship:

$$c_o d_a^2 = f \qquad (3)$$

holds between the average cube edge d_a, the fraction f of the particle surface predisposed to develop into asperities and the maximum concentration c_o with which they tend to emerge on the surface. Then, if the rate of generation of asperities is large in comparison to the rate of change of the particle surface, the following equation expresses the balance of asperities on the particle:

$$\frac{dc}{dt} - k'c_o + k'c + k''c = 0 \qquad (4)$$

where c is the actual asperity concentration and k' and k" the constants of the rate equations relative to the removal of elements by combustion and attrition respectively. The first term of the equation is the rate of accumulation of asperities, the second the rate of their generation, the third and the fourth the rates at which they disappear. The same rate equation constant k' enters into the second and the third term because the removal of elements by combustion, which extends over the entire surface, both generates and consumes asperities. Integrated with the condition that at t=0, c=0, i.e. that asperity concentration after the pretreatment with nitrogen is negligible, Eq. 3 gives:

$$c=c_o\frac{k'}{k'+k''}\,(1-e^{-(k'+k'')t}) \qquad (5)$$

For $t>>0$, $c=c_o\ k'/(k'+k'')$, which reduces to $c=c_o$ and $c=0$ for $k'>>k''$ and $k'<<k''$. Using Eq. 5 and accounting for the change of the particle surface during burn-off by means of Eq. 2 with $a=1/2$, it is:

$$e_c = k''c.N\pi d^2\phi_2^2 \cdot \rho_c\ d_a^{\ 3} =$$

$$= \frac{k'k''}{k'+k''}\,(1-e^{-(k'+k'')t})c_oN\pi d_i^2(1-t/t_{bo})\phi_2^2\ \ \rho_c d_a^3 \qquad (6)$$

where ρ_c is the particle density of coke.

The rate equation constant k' is evaluated by equating the moles of O_2 diffusing to the particle per unit time and the moles of CO_2 corresponding to the mass of carbon of the elements removed per unit time over the whole particle surface by combustion. It is:

$$k'\ \frac{c_o}{f}.\pi\ d^2\phi_2^2.\ \frac{\rho_c d_a^3}{M_c} = \frac{Sh\,D_g}{d\ \phi_1}.\pi d^2\phi_1^2.c_T\ y_{O_2} \qquad (7)$$

where D_g is the oxygen molecular diffusion coefficient in gas phase, c_T the total gas phase molar concentration at the bed temperature, y_{O_2} the oxygen mole fraction, M_c the molecular weight of carbon, and c_o/f the number of all the elements exposed to combustion per unit particle surface. On the other hand, k" is determined considering that, by definition, it is:

$$1-\eta = \frac{k''c}{k''c+k'c_o/f} \approx \frac{k''}{k''+(k''+k')/f} \qquad (8)$$

when taking into account the relationship between c and c_o at t>>0. Values of k' and k" given in Table 1 were obtained from Eq. 7 and 8 by taking into account Eq. 3 and assuming f=0.5. Values of Sh, d_a and η were those given in Table 1. Using such values, calculated attrition rates e_c and specific attrition rates $e_c/N \pi d^2 \phi_2^2$ were determined with Eq. 6 and compared in Figs. 7 and 8 with data from experiments.

With the exception of the exceedingly large $e_c/N \pi d^2 \phi_2^2$ at high burn-off in Fig. 8, the model provides a description of attrition curves. All the variables that it includes were experimentally determined in this work. Actually, some of these variables, like Sherwood numbers, particle burn-out times and particle shrinkage rates, can be predicted on the basis of literature correlations; others, namely the fraction f of surface developing into asperities, the element size d_a and the rate equation constant k" need to be measured or evaluated from experimental data in the present state of knowledge. The rate equation constant k' depends on variables of both these types. Its values have been determined in correspondence of the maximum e_c at t=t' and extended to the entire range $0<t<t_{bo}$. Equation 6 shows that k' significantly affects e_c at low t. In particular, the rate of increase of e_c when switching from nitrogen to oxygen containing gas increases with k' being $(de_c/dt)_{t=0} \propto k'$. Such rate is also proportional to oxygen concentration in fluidizing gas as shown by the substantially constant values of the ratios $k'/(\%O_2)$ in Table 1. Changes of the element size d_a, which influences k' through Eq. 7, have an effect on the location more than on the level of the peak of e_c. Such level is principally determined by k". Table 1 indicates that k" is sensitive to oxygen concentration under mild oxidizing conditions. Changes in the value assumed for f are compensated in the calculations of e_c from the consequent changes of k". Further experimental work carried out by testing carbons of different textures and mechanical properties in beds with different degrees to agitation should be carried out to ascertain possible further implications of the model.

CONCLUSIONS

The comparison between rates of generation of fines when fluidizing batches of petroleum coke under inert and oxidizing conditions confirms that combustion enhances attrition under the experimental conditions tested. A mechanism of surface "activation" characterized by the formation of detachable asperities is responsible for such increase as shown by the microphotographs of cross sections of coke particles submitted to different treatments. Although some internal burning might affect the cortical region of the particle, experiments confirm that combustion of coke essentially occurs according to the shrinking particle model, at least for fractional burn-off below 0.80.

The shape of attrition curves with a peak value of attrition rates somewhere between the beginning of combustion and burn-out is to be related to the opposite effects of surface "activation", which leads to an increased concentration of asperities, and particle shrinkage resulting in the reduction of the surface. Both these phenomena are satisfactorily described by the proposed model. The concept of elements reacting at different rates because of their different accessibility to oxygen is useful to a unified approach to combustion and attrition of coke particles when investigating the mechanism of the generation of fines by abrasion.

NOMENCLATURE

c	number of asperities per unit particle surface
c_o	maximum number of asperities per unit surface
c_T	total gas phase molal concentration
d	average coke particle diameter
d_a	size of elements
D	diameter of volume or surface based equivalent sphere
D_g	oxygen molecular diffusion coefficient in gas phase
e_c	instantaneous carbon attrition rate
f	fraction of coke particle surface developing into asperities
k'	rate equation constant of element removal by combustion
k''	rate equation constant of element removal by attrition
M_c	molecular weight of carbon
N	number of coke particles collected from the bed
Sh	Sherwood number
t	time
t'	time for maximum e_c in attrition curves
t_{bo}	burn-out time
w_c	bed carbon content
y_{O_2}	oxygen mole fraction
$\eta, \bar{\eta}$	carbon combustion efficiency
ξ	fractional burn-off
ε_b	bed voidage
ρ_c	coke particle density
ϕ_1	volume based shape factor
ϕ_2	surface based shape factor

Subscript

i	initial

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CAPTIONS

Fig. 1 - Microphotographs and model representation of particles of petroleum coke.

A - Cross-section of a particle after 60 min treatment in nitrogen fluidized bed

B - Cross-section of a particle after 15 min burning in bed fluidized with nitrogen containing 4.5% O_2

C - Cross-section of a model particle divided into elements reacting at different rates. (The dotted line represents the external particle surface).

Fig. 2 - Ratio of the number of collected to charged particles versus burn-off (A) and fractional burn-out time (B).

Fig. 3 - Fraction of initial particle diameter versus burn-off.

Fig. 4 - Fraction of initial particle diameter as a function of fractional time for complete conversion.

Fig. 5 - Rate of attrited carbon vs time for mechanical and combustion assisted attrition.

Fig. 6 - Change in carbon attrition rates when switching from mechanical to combustion assisted attrition (A,B), and viceversa (B).

Fig. 7 - Attrition rate versus time for different oxygen concentrations in fluidizing gas.

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Fig. 8 - Attrition rate per unit particle external surface versus fractional burn-out time for different oxygen concentrations in inlet gases.

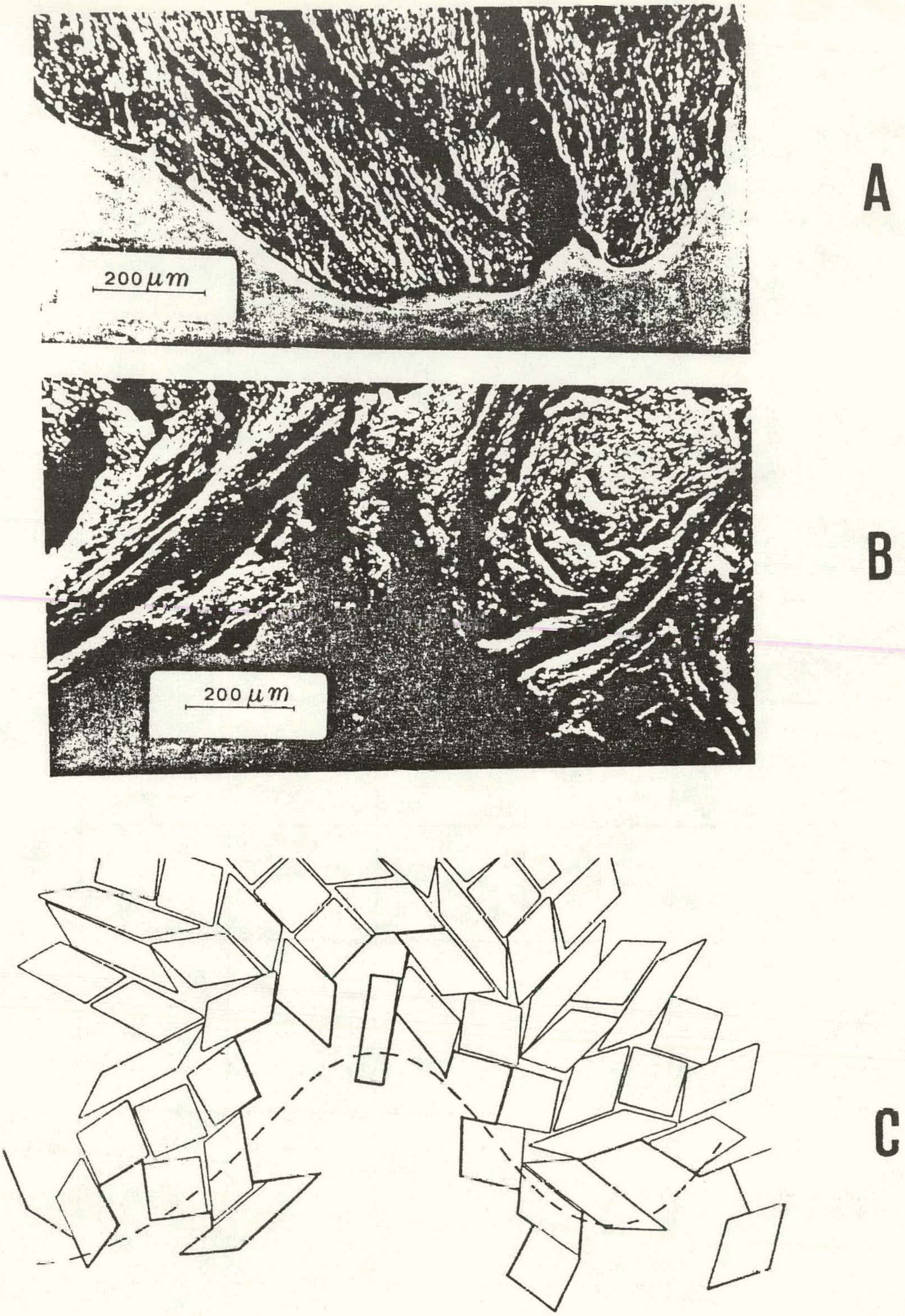
———— lines through experimental points

-------- lines from model calculations

TABLE 1- DATA CHARACTERIZING FLUIDIZED COMBUSTION AND ATTRITION OF PETROLEUM COKE

O_2 Concentration in fluidizing gas	Burn-out time	Time for maximum attrition rate	Coke particle average size(*)	Sherwood number (*)	Combustion efficiency		Size of attrited fines (*)		Rate equation costant for element removal by: (*)		
							Size range	Average size	Combustion		Attrition
									k'	$k'/(O_2\%)$	k"
%(by volume)	t_{bc}, min	t', min	d, mm		$\bar{\eta}$	η (*)	mm	d_a,mm	min^{-1}	min^{-1}	min^{-1}
21	22	5.6	4.69	1.68	0.954	0.951	0 - 0.20	0.11	0.42	0.020	0.048
10	40	10.5	4.61	1.77	0.927	0.922	0 - 0.20	0.11	0.23	0.023	0.046
4.5	80	16.0	4.88	1.98	0.921	0.913	0 - 0.20	0.11	0.10	0.022	0.024
1.4	240	32.0	5.00	2.12	0.897	0.896	0 - 0.20	0.097	0.034	0.024	0.010

(*) Determined at the time t' for maximum attrition rate



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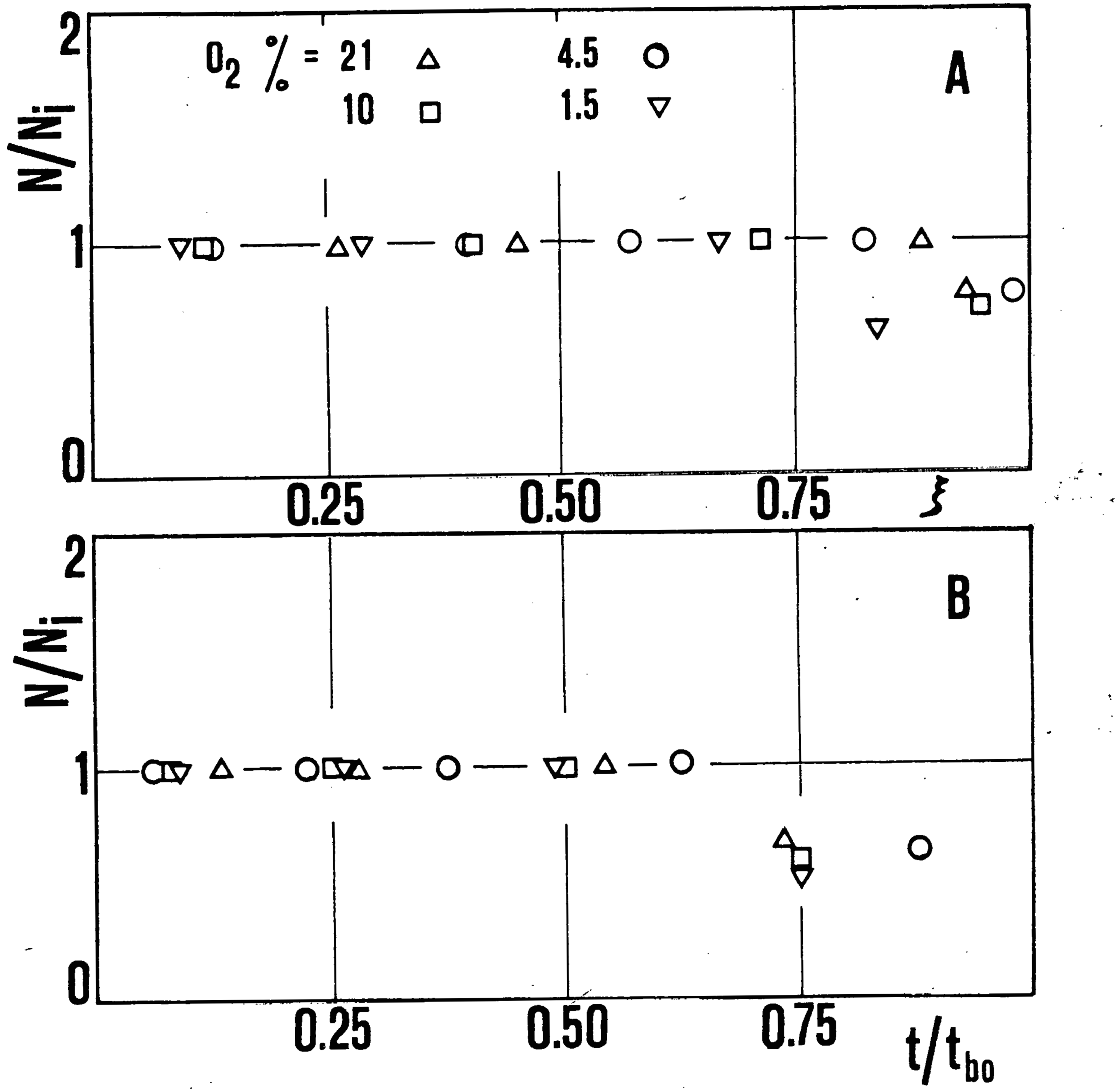


Fig. 2 - Ratio of the number of collected to charged particles versus burn-off (A) and fractional burn-out time (B).

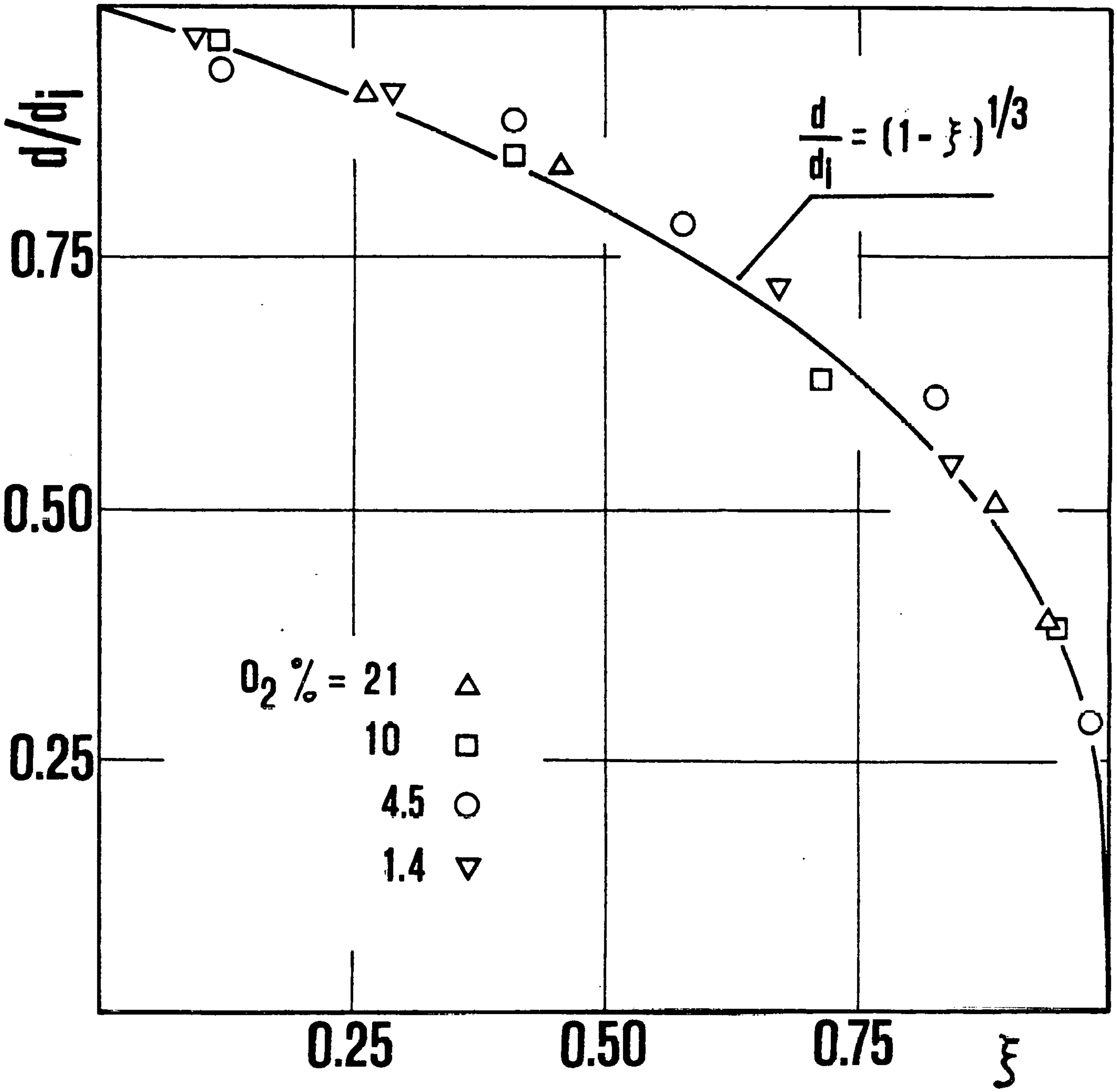


Fig. 3 - Fraction of initial particle diameter versus burn-off.

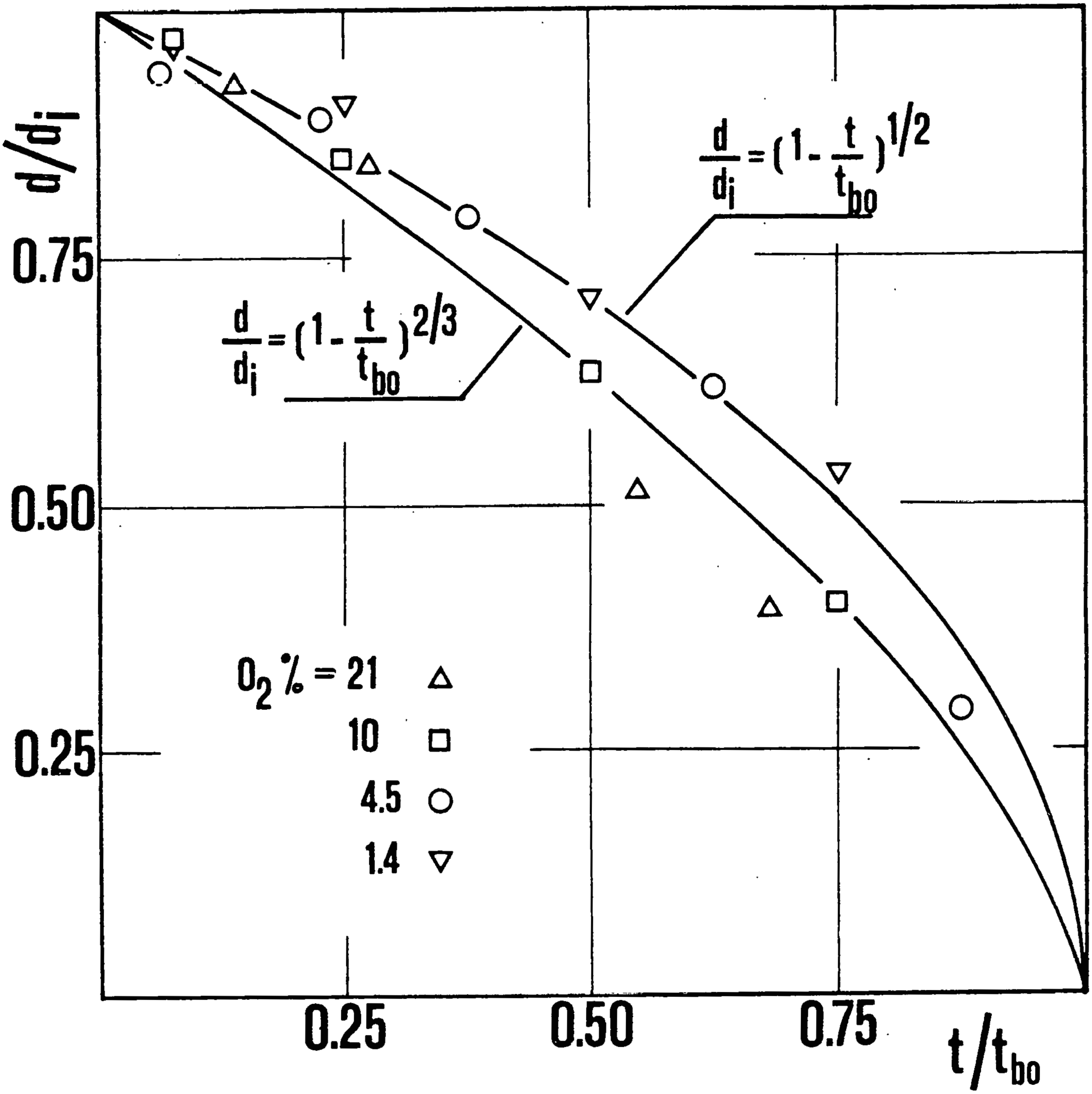
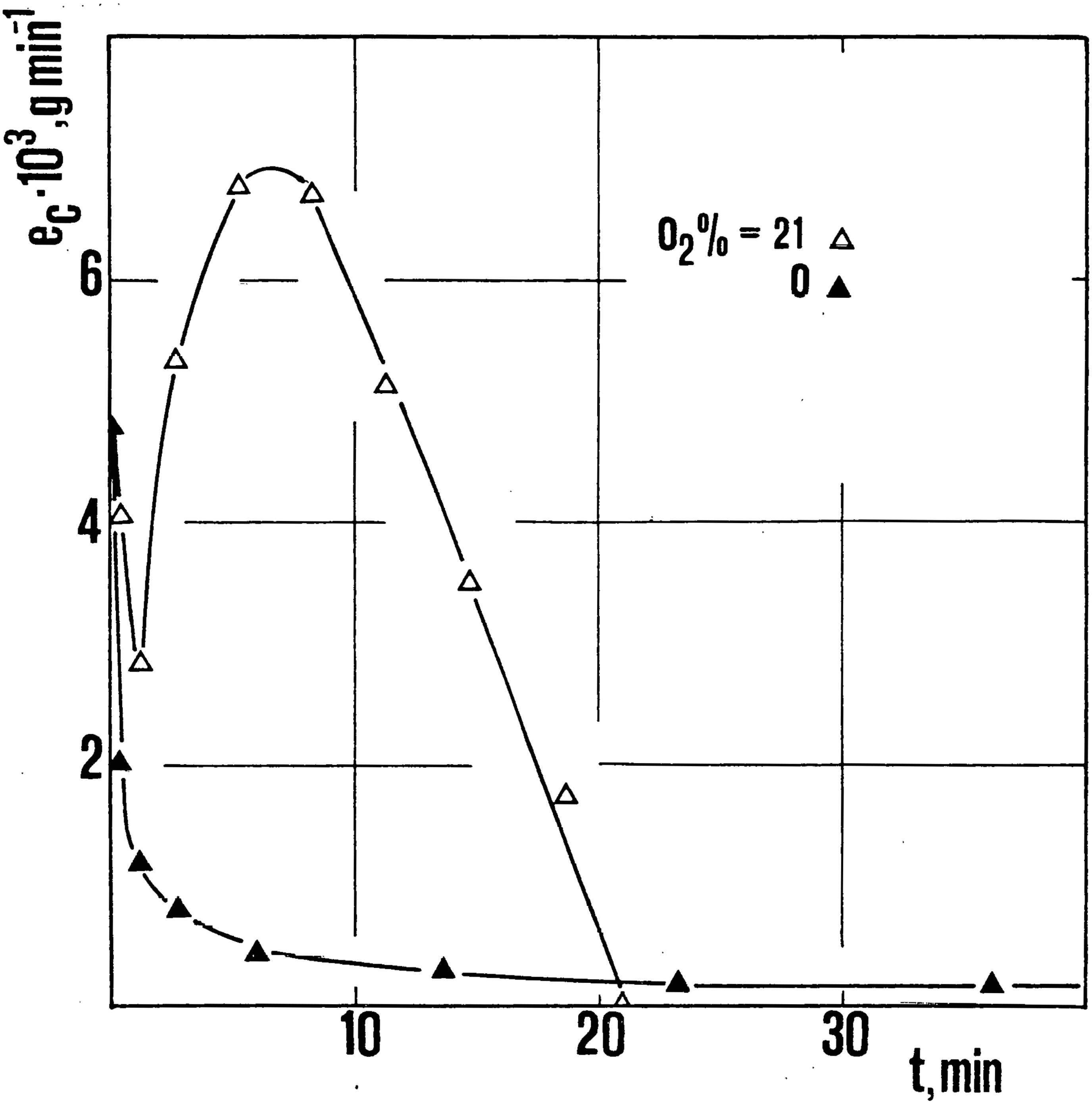


Fig. 4 - Fraction of initial particle diameter as a function of fractional time for complete conversion.



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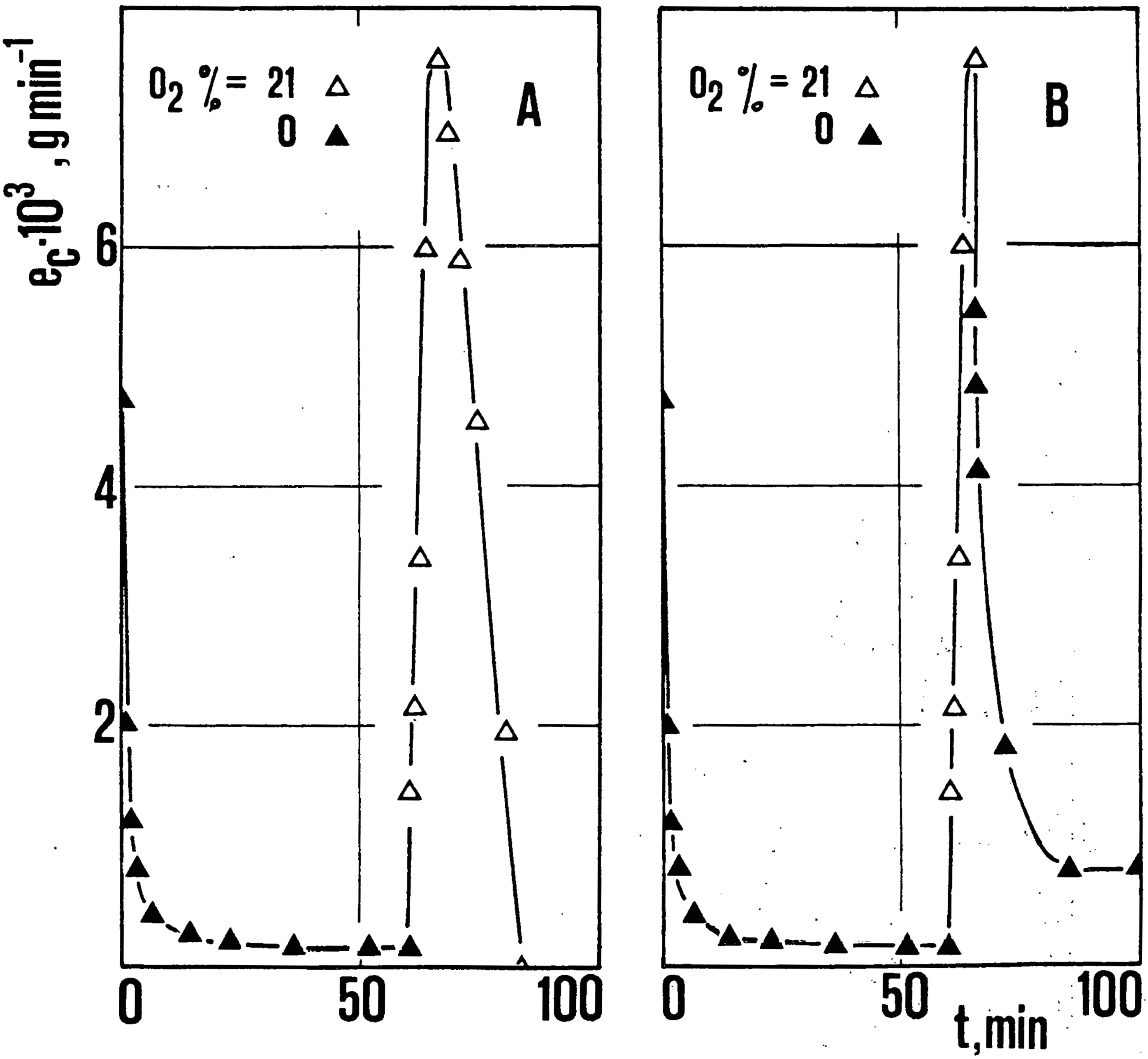

Fig. 6 - Change in carbon attrition rates when switching from mechanical to combustion assisted attrition (A,B), and viceversa (B).

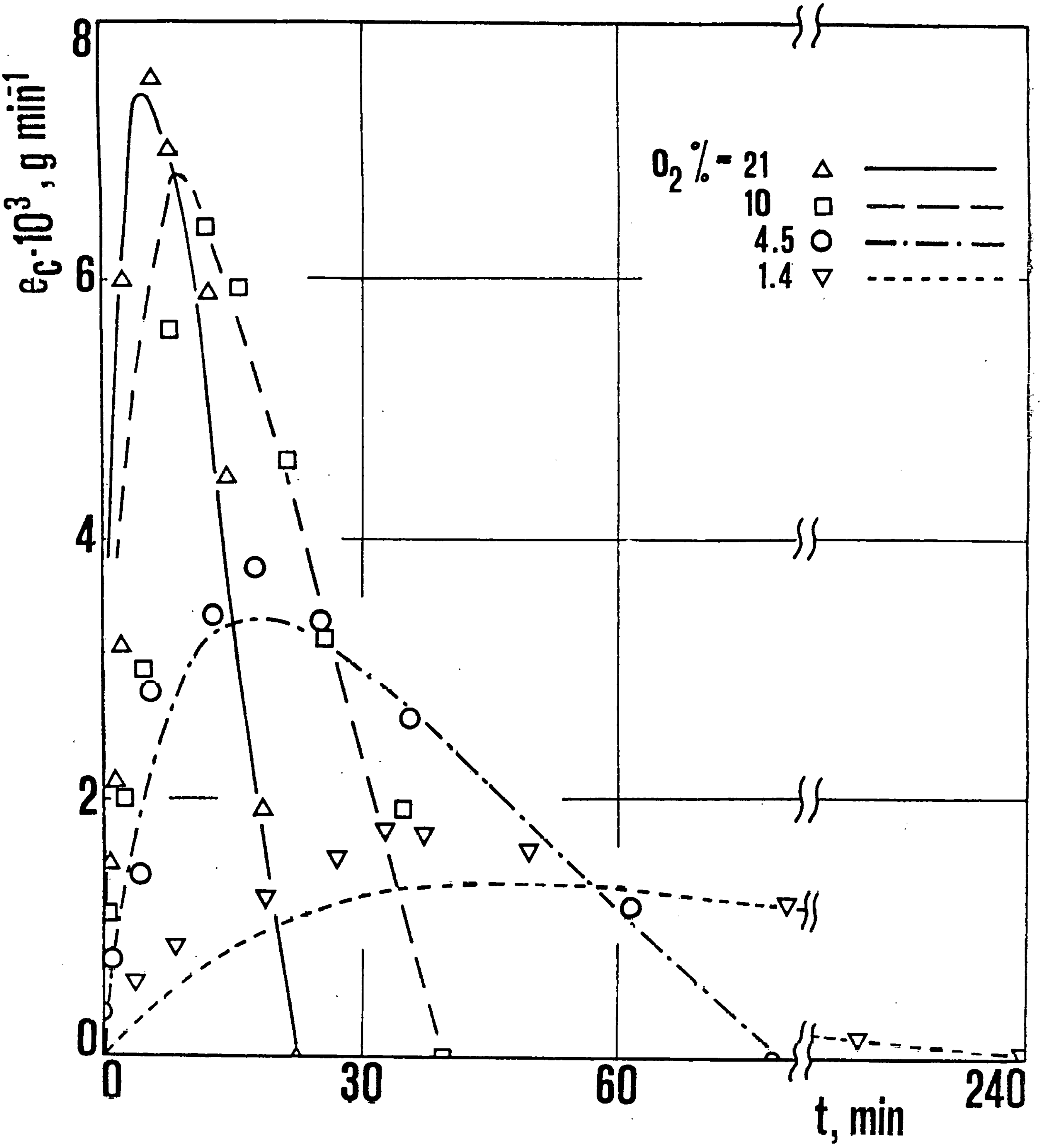

Fig. 7 - Attrition rate versus time for different oxygen concentrations in fluidizing gas.

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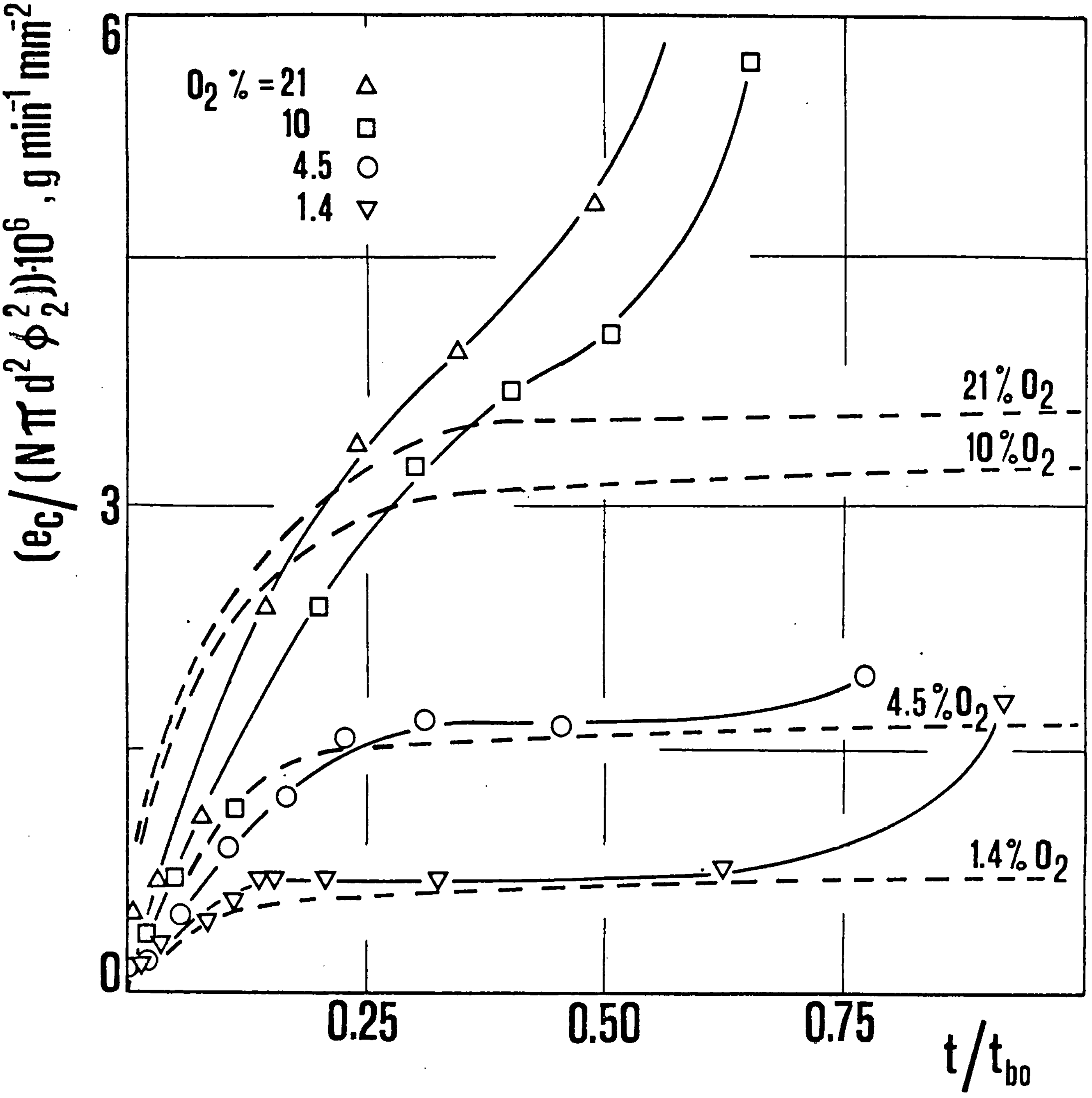

Fig. 8 - Attrition rate per unit particle external surface versus fractional burn-out time for different oxygen concentrations in inlet gases.

——— lines through experimental points

-------- lines from model calculations