RADIATION EFFECTS IN THE ACID HYDROLYSIS OF EUCALYPTUS WOOD AND SUGARCANE BAGASSE

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1. MATERIALS AND METHODS

Diluted acid hydrolysis of either eucalyptus wood or sugarcane bagasse, pre-irradiated at 2×10^5 Gy by electron beam processing (EBP) and crushed to a 50 mesh particle size (1) were performed in a closed batch reactor, using the experimental system illustrated in Fig.1. This system is being used to collect data for determining the rates of the two consecutive reactions governing the saccharification of cellulose: cellulose $\xrightarrow{k_1}$ reducing sugar $\xrightarrow{k_2}$ decomposition products.

Initially, the hydrolysis reactor R is partially filled with a suspension of wood (or sugarcane bagasse) powder in water (about 10% w/v). The suspension is heated and kept at the temperature chosen for each experience (from 165^{o}C to 195^{o}C) by means of an internal electrical heater, automatically controlled. Total effective volume of reactor R is 500 cm^3.

The pressure vessel V_2 is charged with a given volume of H_2SO_4 aqueous solution (about 7% w/w) that will be transferred later, under nitrogen pressure supplied by cylinder C, to reactor R for initiating the hydrolysis reaction. In each case, the volume of acid solution to be transferred was the necessary to obtain, in reactor R, the desired H_2SO_4 concentration for the hydrolysis experiment (between 0,25% and 1.6%).

Before transferring, the acid solution contained in vessel V_2 was heated and kept at the same temperature as in reactor R, using a similar automatically controlled heater. Thus, the hydrolysis reaction will start at the pre-established experimental conditions of temperature and acid dilution and therefore error due to heating delay will be minimized. To stop the reaction at the desired time, a pre-calculated volume of NaOH solution, just enough for neutralizing the hydrolyzated, is also transferred under nitrogen pressure from vessel V_1 to reactor R. In this case, the NaOH solution is at room temperature.

The empty volume originally left in reactor R is enough to accomodate the successive additions of acid and alkali fluids.

The rise on pressure associated with the increased volume of liquid inside the reactor R is counterbalanced through a relief valve, adjusted to open at pressures exceeding 20 kg/cm^2, approximately.

With the system described above, the following hydrolysis conditions were investigated for both, irradiated and non irradiated substrates:

- temperature: from 165°C to 195°C
- acid concentration: 0.25%, 0.5% and 1.6%
- reaction time: from 1 to 360 minutes.

The reducing sugar concentration in the hydrolyzated was determined by the Miller's method, using the dinitrosalicylic acid reagent (DNS).

2. RESULTS

Table 1 summarizes the conditions and results of a preliminary series of runs performed with wood powder at the mean temperatures of 165°C, 175°C and 185°C respectively, keeping the H_2SO_4 concentration at 1.6% in all cases. Each run is composed by several tests with different hydrolysis periods, from 1.5 min.

Based on the results of the preliminary series of runs and considering the economical aspects that should prevail in any industrial application of this process, the bulk of the experimental work was concerned with the hydrolysis at 0.5% H_2SO_4 concentration.

Figures 2 and 3 represent the experimental data obtained in about 400 hydrolysis reactions with eucalyptus wood and 250 with sugarcane bagasse, irradiated and non irradiated, at 165°C and 195°C, with 0.5% H_2SO_4, expressed as mg of reducing sugar per ml of hydrolyzed. This values can be converted in grams of sugar per gram of substrate, multiplying them by the constant factor 0.012.

Degradation of a glucose standard under the same experimental conditions is shown in Fig. 4.

3. CONCLUSIONS AND COMMENTS

The plot of reducing sugar concentration vs hydrolysis time, gave rather scattered points shown in Figs. 2 and 3. This result can be explained by experimental erros due to non uniform distribution of heating through the pulp in reactor R, (particularly at high temperatures), since no forced mixture was present during the hydrolysis period. Another reason may be the natural variations in the structure and composition of the samples.

Nevertheless, the curves interpolated among the experimental points clearly demonstrate that EBP of eucalyptus wood and sugarcane bagasse at the dosage of 2×10^5 Gy, improves the efficiency (and the economics) of the diluted acid hydrolysis process in two ways: it reduces the time to reach a given sugar yield and, for the same reaction period, it increases that yield. About 50% increase of reducing sugar was obtained when irradiated wood and sugarcane bagasse were hydrolyzed with 0.5% H_2SO_4 at 165°C during 70 and 40 minutes, respectively, as compared with native samples. For 195°C and a shorter reaction times (from 20 to 30 min) the effect of radiation on the reducing sugar yield was not so noticeable but still significant (25 to 15%). Further improvments may be achieved by using a percolation process instead of a close reactor and this possibility should be studied.

Table 1. Results of 1.6% H_2SO_4 hydrolysis of wood in a closed reactor system, as a function of time and temperature.

HYDROLYSIS PERIOD (min)	REDUCING SUGAR CONCENTRATION IN THE HYDROLYZATE (mg/ml)			
	2×10^5 Gy IRRADIATED WOOD			NON-IRRADIATED
	RUN 01:165°C	RUN 02:175°C	RUN 03:185°C	RUN 04:165°C
1.5	12.5	13.2	13.9	7.0
3.0	13.9	11.1	13.8	9.2
5.0	14.3	11.1	14.1	10.2
7.0	17.9	13.9	15.0	10.4
10.0	19.0	15.2	15.2	12.4
12.0	18.5	15.1	15.8	12.0
15.0	15.9	16.3	16.4	12.2
20.0	15.0	15.6	16.8	11.0
25.0	16.2	16.0	12.9	10.6
50.0	15.1	14.9	11.0	10.8

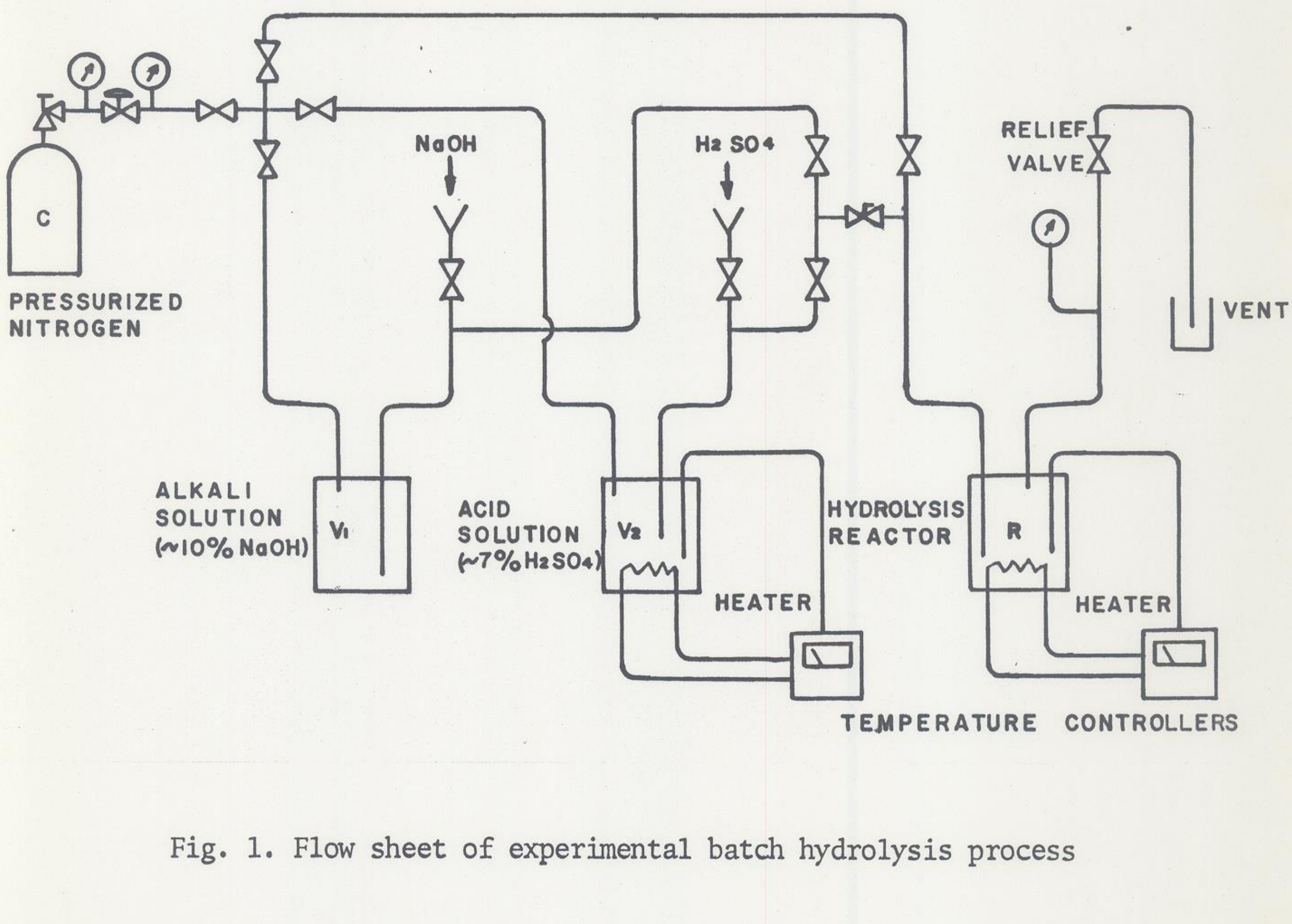

Fig. 1. Flow sheet of experimental batch hydrolysis process

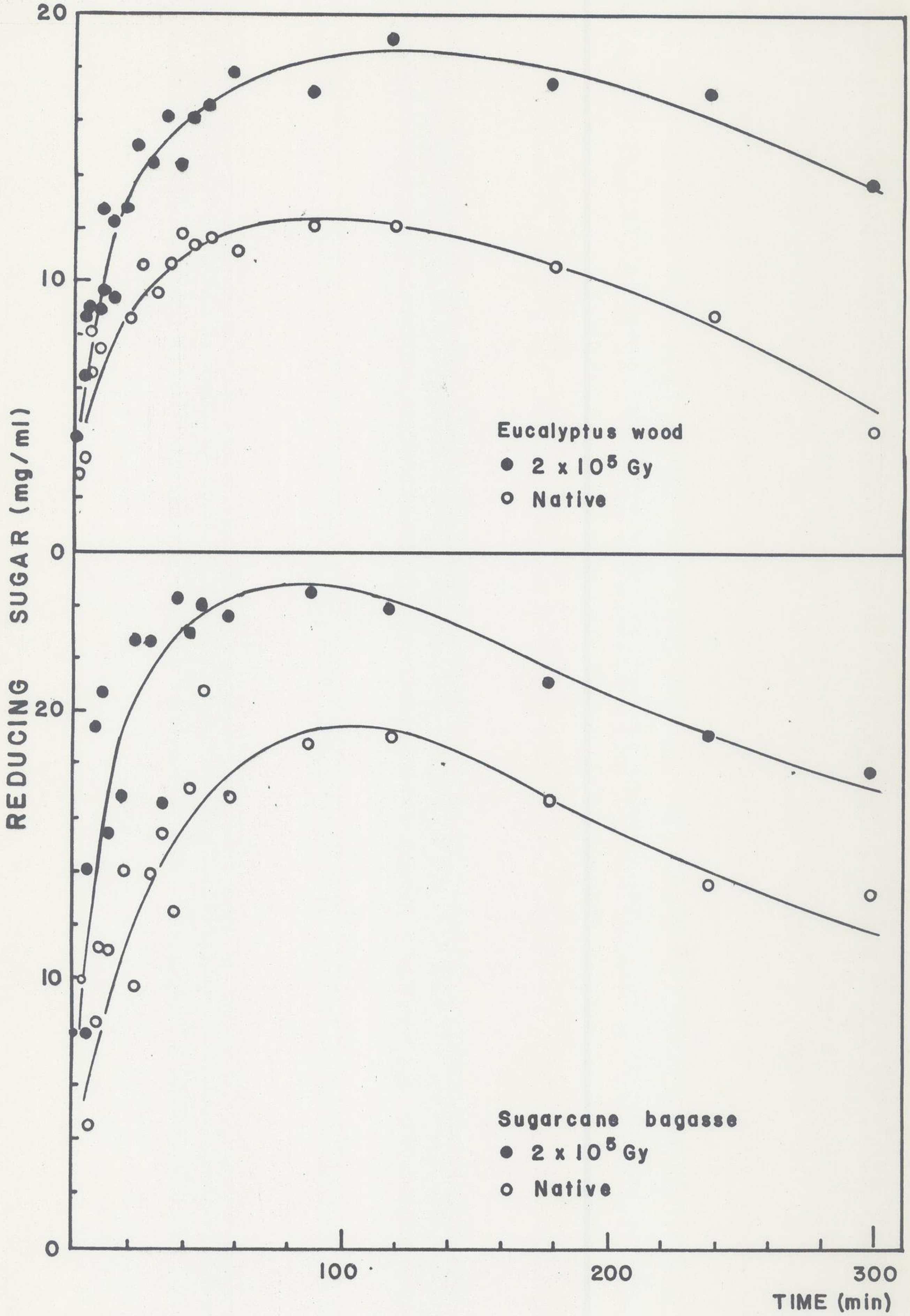

Fig. 2. Reducing sugar concentration as a function of time for hydrolysis at 165°C with 0.5% H_2SO_4.

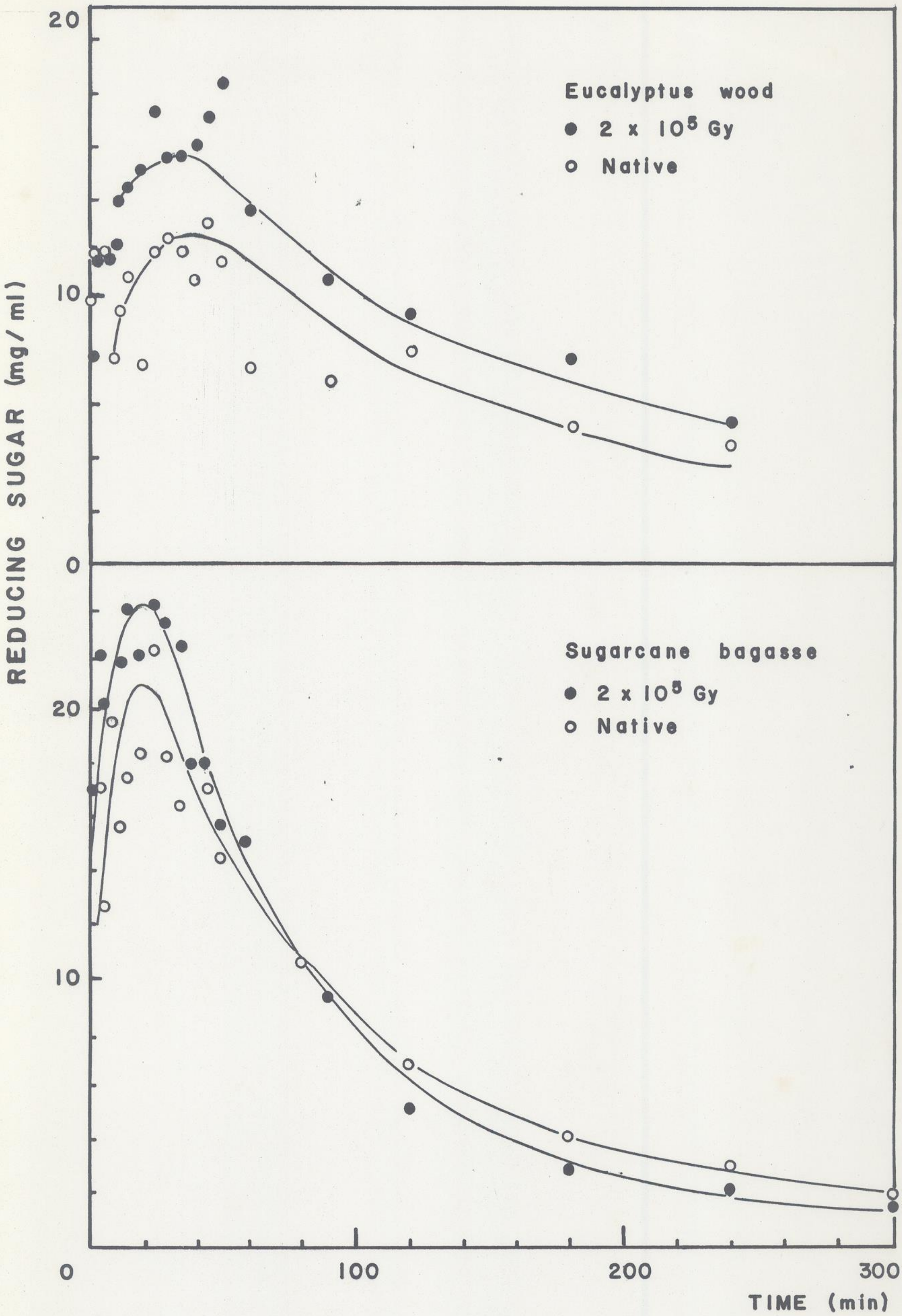

Fig. 3. Reducing sugar concentration as a function of time for hydrolysis at $195^{o}C$ with 0.5% H_2SO_4.

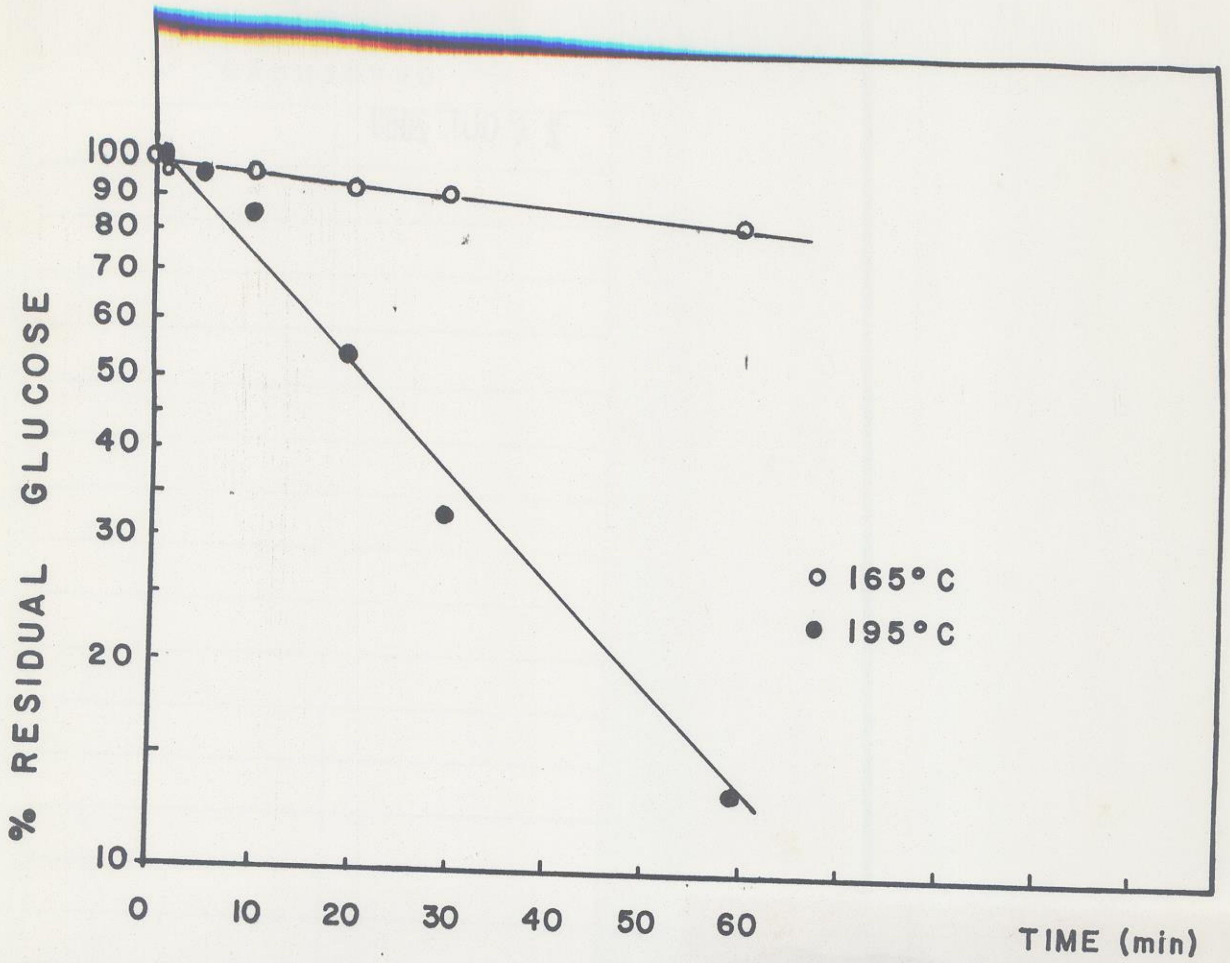

Fig. 4. Degradation of a glucose standard as a function of time, at 165°C and 195°C, using a 0.5% H_2SO_4 aqueous solution in the hydrolysis reactor.

Acknowlegment: This work was partially supported by the International Atomic Energy Agency.

REFERENCES

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