

SPECIFIC RESISTANCE OF REFINERY PROCESS CARBONATATION FILTER CAKE (MUD)

By C. M. YOUNG

Hulett's South African Refineries Ltd.

Introduction

A simple means of measuring the filterability of the Carbonatated Liquor, in practice, at the filter station is required:—

Basically, the instantaneous flow rate of the filtrate is inversely proportional to the specific cake resistance and other factors, and proportional to the pressure and area:

$$\frac{dv}{d\theta} = \frac{PA^2}{\eta(r_1cV + RA)} \quad \dots 1$$

The development of this approach has been covered in Chemical Engineering text books.

Purchas⁽²⁾ states that in this form it is scarcely practical to proceed with tests on *samples* unless one is certain the sample represents the material handled on large scale.

In this exercise, use is made of Carman's equation⁽¹⁾ for constant Pressure conditions.

$$\frac{\theta}{V} = \frac{\eta r_1 c}{2PA^2} V + \frac{\eta R}{PA} \quad \dots 2$$

wherein, the effect of the resistance of the filter cloth and pre-coat is separated from the effect of the resistance of the cake under the controlled test conditions. Carman⁽¹⁾ also warns that the "primary layer of cake should be deposited at low velocity, i.e. at low pressure, since, otherwise the cloth becomes plugged and the initial resistance becomes unduly high."

This form is difficult to use in production, and since we can measure $\frac{dv}{d\theta}$ and θ in Production, the above equation was modified into the following form:— (See Appendix 1.)

$$\left(\frac{d\theta}{dv}\right)^2 = 4\left(\frac{\eta r_1 c}{2PA^2}\right)\theta + \left(\frac{\eta R}{PA}\right)^2 \quad \dots 3$$

and theoretically, if we plot graphically

$$\left(\frac{d\theta}{dv}\right)^2 \text{ against } \theta$$

we should obtain a straight line of slope

$$4\left(\frac{\eta r_1 c}{2PA^2}\right)$$

and hence r_1 since the other terms are readily determined in a well controlled factory.

Thus if a filter in the production line is fitted with a rate indicator, then we should be in a position to measure the specific resistance r_1 at constant pressure, in terms of standard units, where r_1 is an inverse function of the filtration characteristics of the carbonatated liquor, as *produced and filtered in the factory*.

Method

The filter used for the tests (in parallel with the production filters) was fed by a centrifugal pump and the inlet pressure controlled. The flow rate out of the filter was measured by means of a flow rate indicator. Time and temperature were noted and samples of the Carbonatated Liquor and filtrate were collected for analysis.

For the exercise it was assumed that the Calcium Carbonate precipitate was incompressible.

Discussion

Initially, the pressure control valve was too large, and later, when a smaller (gate) valve was fitted control of pressure was also found to be difficult. However, sufficient tests were carried out to show the merit of the system, and no doubt with a feed pump of good characteristics and possibly an automated pressure control system, we may arrive at a system where three readings will be sufficient for an operator to record the filterability due to any changes he may make to the process. The system may possibly be enlarged to dictate the economic level for changing filter cloth, milk of lime addition and so on.

It should be noted, however, that any error in measuring $\frac{d\theta}{dv}$ is squared.

Summary

A simple method of determining the filterability of Carbonatated Liquor in terms of the specific resistance of the filter cake (r_1) is given for constant pressure conditions.

Results of tests, under rather crude conditions, show merit for the system, and using readily available control systems, this method could be used to measure the effect of process changes.

Symbols⁽¹⁾:

V = cm³ of filtrate in time θ secs.

$\frac{dv}{d\theta}$ = rate of flow of filtrate, in cm³/sec.

TABLE II

Date	Test number	Pressure	Brix	Temp.	Viscosity	Insoluble solids concentration	Filter area	Slope of curve	Specific resistance	Reciprocal of r_1	Laboratory filterability	Work done per filter cycle
		$P \times 10^3$			$\eta \times 10^{-2}$	$c \times 10^{-3}$	$A \times 10^5$	$m \times 10^{-10}$	$r_1 \times 10^8$	$\frac{1}{r_1} \times 10^{-9}$	F	W.D.
		Grams/cm ²		°C	Poises	Grams/cm ³	cm ²	Sec/cm ⁶	Sec ² /Gram	Grams/Sec ²	%	Tons filtered as sugar
29. 9.66	5	3.0	64.8	88.0	7.6	11.1	2	2.4	0.7	14	34	68
30. 9.66	6	3.0	62.7	86.0	6.1	9.6	2	3.8	1.6	6	24	18
7.11.66	8 (a)	2.5	64.8	82.2	8.0	10.7	2	0.7	0.2	50	31	17
7.11.66	8 (b)	5.0	64.8	82.2	8.0	10.7	2	1.1	0.5	20	31	17
8.11.66	9 (a)	3.0	64.0	82.2	7.3	10.7	2	0.6	0.2	50	33	26
8.11.66	9 (b)	3.0	64.0	82.2	7.3	10.7	2	0.9	0.3	33	33	26
9.11.66	10	3.0	63.5	82.2	6.9	11.9	2	5.2	1.5	7	25	20
22.11.66	12	3.0	63.7	79.4	7.8	10.9	2	1.0	0.3	33	—	42
23.11.66	13	2.5	62.7	79.4	7.0	9.1	2	3.4	1.1	9	37	25
6.12.66	17	4.0	60.9	79.4	5.8	9.0	2	4.3	2.6	4	28	19
9.12.66	18 (a)	1.0	63.3	77.8	7.8	9.4	2	8.0	0.9	11	33	42
9.12.66	18 (b)	3.0	64.5	80.3	8.2	9.4	2	1.5	0.5	20	33	31
12.12.66	19 (a)	1.0	62.2	78.3	6.8	9.1	2	7.8	1.0	10	33	32
12.12.66	19 (b)	3.0	63.4	81.7	7.0	12.3	2	1.7	0.5	20	33	32

Worked Example:

Test No. 9(a): $\left(\frac{d\theta}{dv}\right)^2$ plotted graphically against θ : Slope of curve = $4m = 2.2 \times 10^{-10}$ secs/cm⁶; $m = 0.6 \times 10^{-10}$ secs/cm⁶ = $\frac{\eta r_1 c}{2PA^2}$.

Hence $r_1 = 0.2 \times 10^8$ sec²/gram. To convert r_1 to chemical engineering units, multiply by the gravitational constant (g) and change units.

e.g. $r_1 = (0.2 \times 10^8) \times 14.6 \times 10^3$ ft/lb = 2.9×10^{11} ft/lb.

Work done per filter cycle

Refers to the average work potential per filter cycle at the main production line.

i.e. W.D. = $M \times S$; where M = Av. melt/hour, in tons; and S = Average filter "start", in hours.

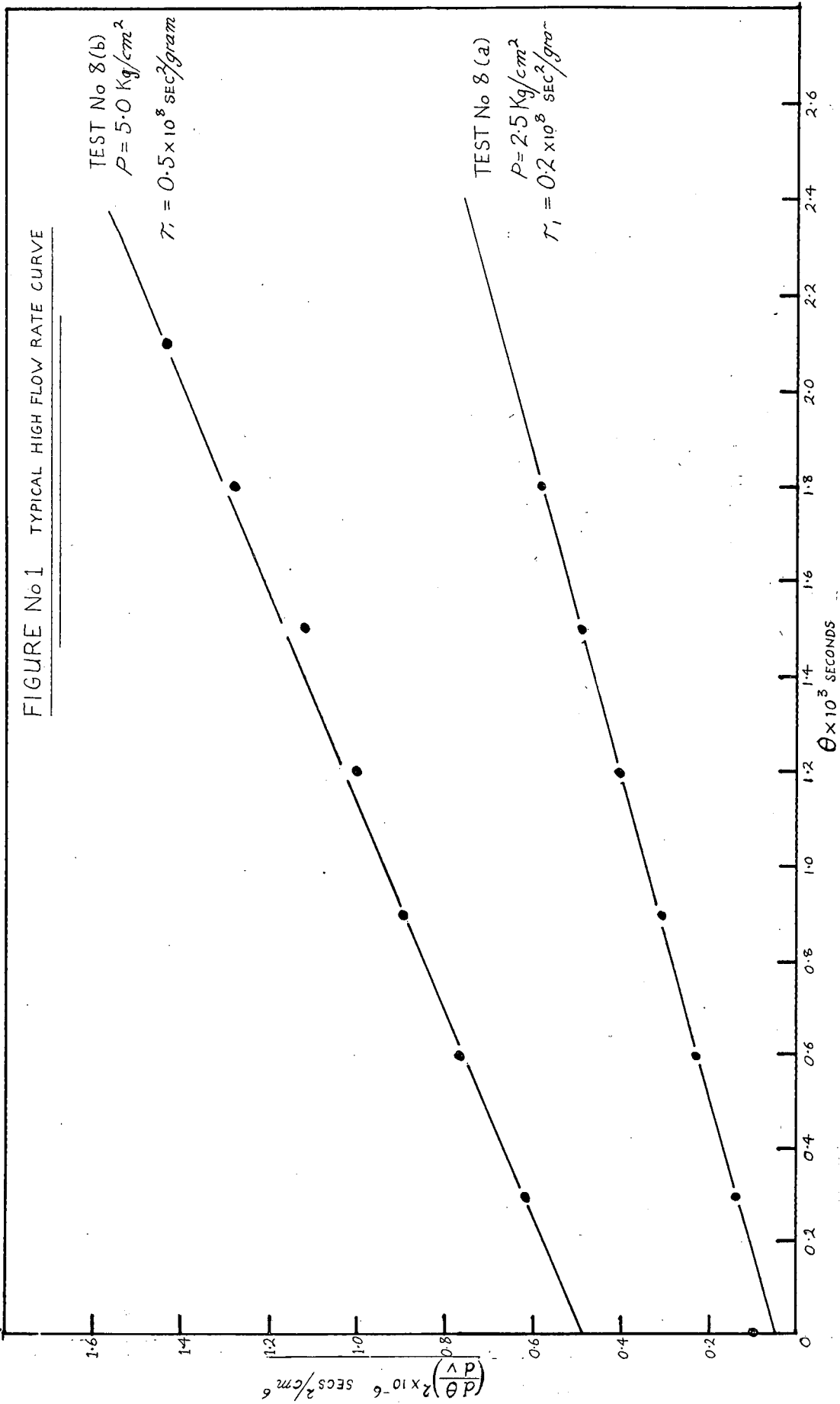


FIGURE No 2 (TYPICAL LOW FLOW RATE CURVE)

TEST No 5 AT PRESSURE $P = 3 \text{ Kg/cm}^2$

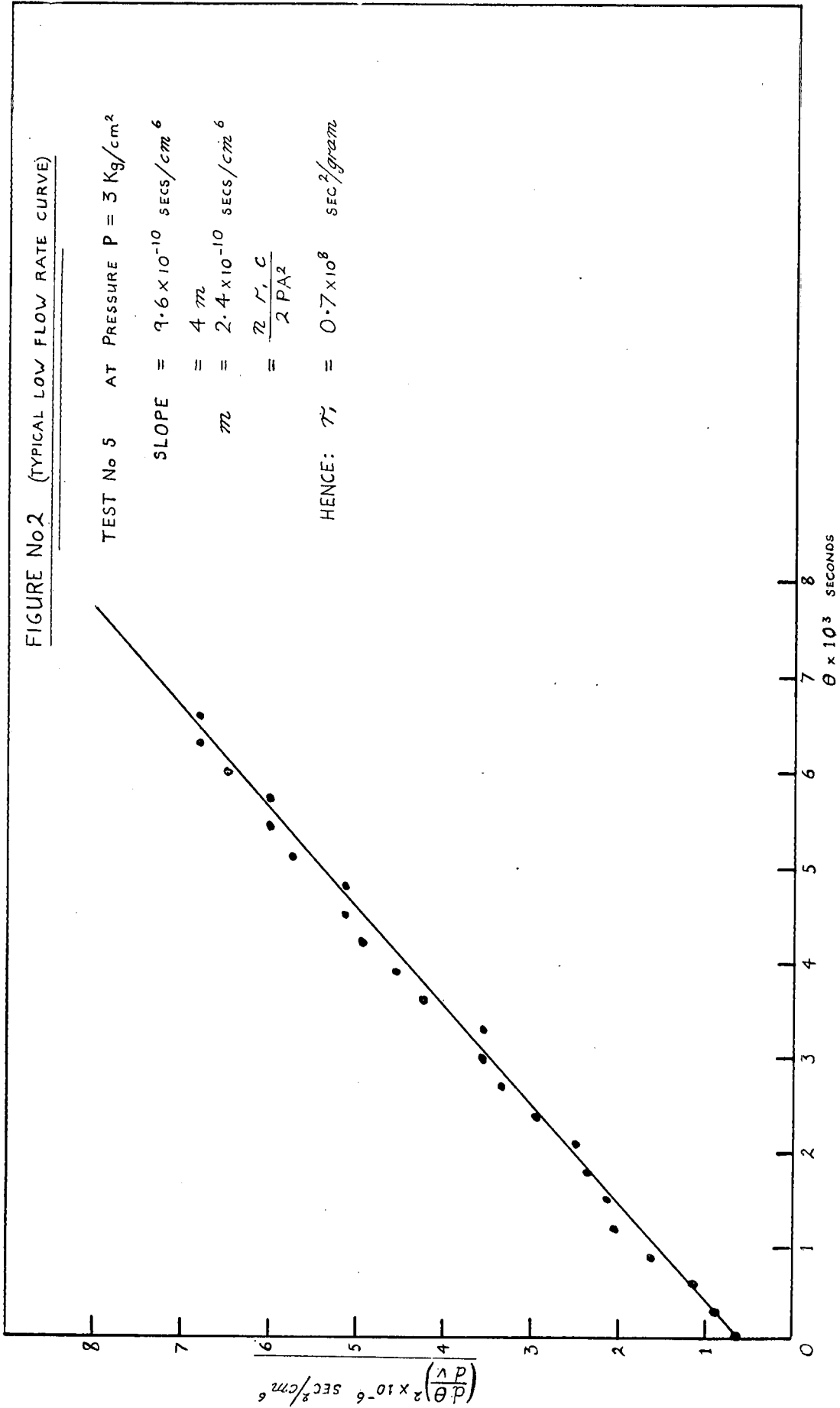
$$\text{SLOPE} = 1.6 \times 10^{-10} \text{ SECS/cm}^6$$

$$= 4 \text{ } \eta$$

$$\eta = 2.4 \times 10^{-10} \text{ SECS/cm}^6$$

$$= \frac{\eta \cdot r, C}{2 \text{ PA}^2}$$

$$\text{HENCE: } \eta = 0.7 \times 10^8 \text{ SEC}^2/\text{GRAM}$$



Discussion

Mr. Young: Referring to the tables in the appendices, it is the data items which are multiplied by the factors as given. To be mathematically correct, the exponents, as printed, should have the signs changed. In the text, R = Initial resistance per 1 cm^2 of filtering surface.

Dr. Douwes Dekker: Mr. Young used a centrifugal pump for pressure in his experiments and it might have damaged the precipitate and affected its specific resistance.

In the last column of Table II, work done per filter cycle is given as tons filtered as sugar. What is the purpose of these figures? There seems to be no correlation between them and the specific resistance of the cake.

Mr. Young: The pump used was oversized, which would give an even more adverse effect.

My quantities for specific resistance are about ten times greater than expected, possibly because of the pump and restriction in valves.

Work done per filter cycle, in Table II, was inserted to give a measure of the performance in the factory. This work becomes directly proportional to filterability only under unique conditions. I can see no correlation with any of the data in Table II. The purpose of this paper is to establish a means of measuring filterability. Further investigations on correlation and significance should be carried out.

Mr. Alexander: I think the filtering quality of the sugars might have had something to do with the high specific resistance figures. Overseas refiners who use calcium carbonate filter cake have come to the conclusion that when they refine Natal raws the resistance does increase.

Mr. Young: It is difficult, at this stage, to take into account the raw sugar filterability. When sugar arrives in the factory and goes through processing, the method by which it is treated has a bearing on its filterability. The churning in the pump does not account wholly for the high specific resistance.

Dr. Matic: A specific resistance of cake is measured and there is constant pressure. Why cannot these be correlated with the behaviour of the sugar in the refinery?

I know that the permeability coefficient of slime dump material has been successfully measured on the gold mines, using Carman's method. Owing to a difference in particle size there was some difficulty in packing the bed uniformly but when this was achieved the filtration of the bed was constant.

Mr. Young: The filterability in the factory can be assessed by the specific resistance of the filter cake for any particular carbonated sugar liquor coming

into the process but at this stage we have no correlation with the filterability of raw sugar as such. For instance, the raw sugar solution has not been carbonated.

Mr. Robinson: Why did Mr. Young plot the square of the differential on his specific resistance graph? The result would have been the same.

Referring to what Dr. Matic said, the specific resistance is a function of porosity and particle density and is given as a figure of surface area per volume.

In a test carried out at the refinery to test the effect of starch, we added starch to a sugar solution, carbonated it and found that with a high starch concentration the particles settled more slowly, indicating they were much smaller. Specific resistance depends very much on particle size. Possibly impurities in the sugar prevent the formation of reasonable size particles.

Mr. Young: If in the original form you plot $dv/d\theta$ against v , the difficulty is in measuring v .

If you look at the equation in Appendix I, $(d\theta/dv)^2$ is plotted against θ , thereby eliminating the volume.

If particle sizes get small they become colloidal and the floc formed is such that the cake is no longer incompressible and other factors come into account.

The specific resistance r_1 is the resistance per 1 gm/cm^2 of dry cake solids. The true specific resistance is the resistance per cm^3 of cake as collected. It is not easy to measure volume of cake but the dry material can be measured. The relationship between the two is given by $r_1 c = rv$. I have used Carman's advocated specific resistance r_1 as it is the one used in most chemical engineering text books.

Dr. Matic: The impurities in a sugar will determine the type of cake and its porosity and that is what we should try to measure.

The particles produced are not incompressible and, according to Dr. Bennett of Tate and Lyle, our sugar is such that the calcium carbonate precipitate formed is different, when viewed under a microscope, from any other sugar. The reason for this is not certain.

Mr. Dedekind: In Table II, the first test was done on 29th September and the figures are rather interesting. Was this sugar fresh, or had it been stored and are any figures available as to its starch content? It has been stated that filterability of a sugar improves with storage.

Mr. Young: I do not have the figures for starch; however, all data is on record at the factory. The purpose of this paper is to establish a method and then subsequently we can investigate the effect of various conditions and constituents on filterability.