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Canada Centre for Mineral and Energy Technology
Centre canadien de la technologie des minéraux et de l'énergie

A NOVEL LABORATORY METHOD TO STUDY DISPLACEMENT FROM PACKED BEDS OF OIL SAND

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July 1986

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Submitted for publication in the AOSTRA Journal of Research

ENERGY RESEARCH PROGRAM
ENERGY RESEARCH LABORATORIES
DIVISION REPORT ERP/ERL 86-37(J)

ABSTRACT

A rapid, inexpensive method has been developed to model the recovery of bitumen from packed beds of oil sand. Excluding the reconstitution and analysis stages, a displacement experiment can be performed in about three hours. Oil sand batches are reconstituted by mixing predetermined amounts of sand, water and bitumen, thus introducing the ability to control each component of a sample. Dilution of the bitumen with hexadecane permits experiments at low temperatures while maintaining an oil-water viscosity ratio comparable to that at high temperatures. It has been observed that the displacement efficiency depends on the oil sand pack density and on the nature of the displacing liquid, i.e. caustic or distilled water. The initial water saturation influences displacement by water, but has little or no effect on caustic displacement. The model shows promise as a tool to investigate the effect of chemical species in oil sand on the displacement operation, thereby assisting in better defining the mechanisms of the recovery process.

INTRODUCTION

As reserves of conventional oil in Western Canada are depleted, Canada will have to rely on its frontier areas and resources of bitumen and heavy oil to supply much of its petroleum demand. Economic development of in situ processes for displacing bitumen from oil sands represents the largest potential source of petroleum products.

Research has been conducted for a number of years and sophisticated models have evolved for predicting field behaviour based on laboratory studies [1,2,3]. The experimental program described in this paper is directed toward identification of the important parameters affecting the displacement process. It was necessary to develop a procedure to allow rapid, inexpensive testing of a large number of experimental conditions to screen those which show promise for more extensive consideration. This paper outlines the development of the experimental test method and presents the initial screening results. Experiments using this physical model to assess the impact on recovery of chemical species residing in oil sand will be detailed in subsequent communication. It is believed that this largely unaddressed approach will be useful for explaining and predicting results obtained in larger models and ultimately in the field.

EXPERIMENTAL

Reconstitution. The objective of this research program is to introduce perturbations to the oil sand composition and to perform displacement tests to evaluate their effect on recovery. Specifically, the characteristics of the mineral matter, the connate aqueous phase and the bituminous phase will be controlled to assess the important chemical interactions influencing the displacement process.

The experimental technique which introduces the ability to control the oil sand composition is termed reconstitution. Clean, toluene-extracted sand from the Athabasca deposit and the desired aqueous phase are mixed by mechanical tumbling interspersed with manual stirring. After approximately one day, the organic phase is introduced and the procedure is repeated. The components are added in predetermined weight ratios to simulate the saturations in medium grade Athabasca oil sand.

Materials. The mineral matter used in reconstitution was obtained by Dean Stark toluene reflux (basically following the procedure developed at the Alberta Research Council [4]) of a sample of medium grade Athabasca oil sand received from the Alberta Oil Sands Sample Bank. Table 1 provides an analysis of its components.

In most cases, distilled water was used in reconstitution. To simulate connate water, reagent grade calcium chloride or sodium chloride was dissolved in the water prior to mixing. The aqueous displacing phase consisted of either distilled water or a solution of sodium hydroxide.

The bituminous phase was prepared by diluting a sample of Suncor bitumen with certified grade hexadecane. The diluent is added to reduce the viscosity of the bitumen in order that:

1. The viscosity-lowered organic phase is more amenable to reconstitution.

2. Displacement experiments can be conducted at low temperatures

while maintaining an oil-water viscosity ratio similar to that existing at elevated temperatures during steam/hot water displacement of bitumen.

The following observations have been made with regard to this mixed organic phase:

1. The agreement between the measured densities of hexadecane-bitumen mixtures and those calculated by assuming ideal mixing (knowing the component densities) is within 0.5%.

2. The viscosities of mixed solutions can be calculated to within 20% of measured values using Cragoe's empirical correlation [5] as shown in Figure 1. This level of agreement is regarded as good because the viscosity of the bitumen changes by orders of magnitude over the temperature range shown.

3. The Dean Stark extraction process has been performed using known amounts of hexadecane and bitumen with the analyzed weights within ±2% of the original weights. The relatively low vapour pressure of hexadecane precludes it being preferentially volatilized at the temperatures encountered in the displacement cell or the extraction apparatus.

4. The asphaltene content of the residual oil in a pellet and the oil produced from a displacement experiment are equivalent, hence the displacement process does not cause a chromatographic separation on the basis of molecular weight.

5. After standing for several months there is no visible segregation due to density differences.

6. Being relatively inert, the hexadecane should not chemically influence displacement by water or caustic.

Packing. Typically 40 g of reconstituted oil sand is packed into the displacement cell (2.54 cm I.D.) using hydraulic pressure. Figure 2 shows higher packing densities result if a given pressure is repeatedly cycled on and off as opposed to continual application. Figure 3 shows that the application of increased pressure causes the extent of sand grain crushing to increase. Simultaneously the amount of oil and water which is squeezed out of the press increases. Therefore it is desirable to use cycling with minimal pressure to achieve the required density. The oil sand was packed to a density of 2.00 g/cm^3 in most experments. This packing procedure is a rapid (about twenty minutes), reproducible method for preparing oil sand samples which have densities similar to those in a reservoir.

Displacement. After oil sand compaction, glass beads are added to occupy the remaining space in the displacement cell and the end caps are secured. The cell is mounted in the apparatus, shown in Figure 4, and a displacement experiment is conducted in the following manner. The cell and inlet/outlet lines are evacuated and the displacing liquid admitted to the system. This displacing liquid is drawn into the cell from both ends so that cross pellet flow is minimized. When filled, the oil sand pellet is in the central part of the cell with all of the void space occupied by the displacing liquid. Heat is applied to the cell through a surrounding jacket until the system reaches thermal equilibrium. Inlet and outlet valves are left open to accommodate thermal expansion during heating. When steady-state conditions are reached the pump is turned on and distilled water is delivered to the cell, forcing the displacing liquid through the pellet and out the collection port. After a predetermined volume of water has been pumped in, heating is stopped and the system is left to cool. The oil sand pellet is removed and a Dean Stark toluene reflux is conducted to measure the amount of residual oil. This permits calculation of the amount of produced oil by difference. Mass balances on the produced and residual oil accounted for 98% of the original oil. The residual oil content was selected as the quantity to be determined for experimental expediency. The Alberta Research Council Dean Stark procedure [4] was modified to allow evaporation of the toluene at room temperature using convective air flow. Evaporation is continued until successive weight readings taken each hour agree within 1%.

RESULTS AND DISCUSSION

Operational variables. Parameters external to the sand were examined to determine the sensitivity of the displacement process to each of the variables. The parameters in Table 2 were varied over the range shown using a two-level fractional factorial design.

A standard deviation of 5.2 was calculated from nine centrepoint repeats using different reconstituted oil sand samples. A standard deviation of 1.1 was determined from four repeats using the same oil sand sample. Therefore, tests performed on a given batch of oil sand are much more discriminating than those performed on different reconstituted oil sands. The experiments in this design involved different samples of oil sand hence the standard deviation of 5.2 applies, and only parameter estimates of absolute value greater than 3.1 are significant at the 95% level. The only significant variable at this level is the nature of the displacing phase. The main effect coefficient associated with this variable is 8.08. All other main effects and all interaction term coefficients had an absolute value less than 3.1 and were therefore regarded as insignificant. As long as the other variables are maintained within the test limits they will not exert a discernible effect on the ultimate recovery.

Additional experiments were performed to extend the range of flowrate test conditions because even with no flow, some of the bituminous phase is displaced from the pellet, presumably by a mechanism of imbibition. As Figure 5 shows, beyond a certain minimum value, flowrate can be varied by as much as an order of magnitude without strongly influencing the recovery.

The density to which the oil sand pellet was packed was studied independently. Figure 6 demonstrates that the pack density is an important consideration in displacement by water, but in displacement by caustic its effect is negligible. As the pellet density increases the principal change to the oil sand occurs in the geometry of the flow channels. At higher density the pores are of smaller diameter and capillary forces are larger. The results appear to indicate that this change in capillary forces causes a change in displacement efficiency by water, but does not affect caustic displacement. This is the first indication of a fundamental difference between displacement by caustic and displacement by water. Although recovery is sensitive to pack density for water displacement, the pellet formation procedure produces densities reproducible to within 0.5% and any shifts in recovery due to changes of this order will be minimal.

It is interesting to note that temperature did not exert a significant effect. Figure 1 indicates that the viscosity of 20%

hexadecane in bitumen varies by nearly an order of magnitude between 30 and 60°C, but recovery was not affected. Table 3 lists the operating conditions selected based on these experiments.

Compositional variables. A second two-level fractional factorial design was performed on six variables related to the internal composition of the oil sand. These compositional variables are listed in Table 4 along with their upper and lower bounds.

The sand drying temperature was selected because after heating at 300°C the sand is immediately water-wet whereas after treatment at 25°C the sand is initially toluene-wet, but seems to revert to a water-wet state after tumbling with water during reconstitution. The sand size distribution and specific addition of kaolin were intended to alter the geometry and pore size distribution in the sand pellet. The hexadecane dilution factor was included to ascertain the effect, if any, of this modification of the bituminous phase. There are potential chemical and viscosity effects in this variable. The connate water content and composition were varied because the water phase is reported to reside between the bitumen and sand surfaces thereby playing an important role in the separation and displacement processes [6,7].

From the design analysis, the displacing phase is again seen to influence the recovery most strongly, however, the effect of the connate water saturation is also significant at the 95% confidence level.

The water content was varied between 0 and 7.8% by weight of the oil sand. This corresponds to initial water saturations from 0 to 43% of the pore volume based on a pack density of 2.00 g/cm^3. In each case, the amount of oil added is adjusted to maintain a constant overall liquid volume in the oil sand samples. Therefore, the initial oil saturation varied between 91 and 48% of the pore volume in these experiments. As shown in Figure 7, the water saturation is a significant factor in displacement by water, but displacement by caustic is largely independent of initial saturation conditions. This is a second indicator of a fundamental difference between displacement by caustic and water. The difference is thought to be related to a stronger sand wetting tendency of the caustic. In the presence or absence of connate water, caustic is able to displace bitumen from the porous medium with about the same efficiency. For water displacement, efficient recovery requires a certain minimum level of water saturation.

The displacement efficiency can be expressed in terms of a percentage of initial oil recovered or in terms of the residual oil saturation remaining. These two different representations of the same data are compared in Figures 7 and 8. Although the shapes of the two sets of curves are different, in both cases there is an abrupt change in recovery between saturations of 1.0 and 3.4% for water displacement only.

The effect of displacing phase pH has been studied by varying the concentration of sodium hydroxide. The results in Figure 9 show that the selected concentration of 0.1 M NaOH (pH 13) represents a near optimal value at which the difference between recoveries using caustic or water is maximized.

CONCLUSIONS

1. The reconstitution technique has been used to prepare batches of oil sand in which the characteristics of each of the component parts can be controlled.

2. Dilution of the bitumen with hexadecane, prior to reconstitution, has simplified the experimental procedure without radically changing the chemical properties of the oil sand system.

3. Pressure cycling has been used to reproducibly pack the oil sand to densities of 2.00 g/cm^3 while minimizing both the extent of sand grain crushing and the amount of liquid squeezed out of the pellet.

4. The displacement test is a rapid, inexpensive method which is discriminating to changes in certain parameters. The basicity of the displacing phase and the connate water saturation introduced during reconstitution exert significant effects on the displacement process.

5. The density of compacted oil sand pellets influences the recovery efficiency for displacement by water, but not for displacement by caustic.

6. Variation of the bituminous phase viscosity by as much as an order of magnitude does not significantly affect the recovery in a displacement test.

7. The amount of water added during reconstitution influences recovery by water, but has negligible effect on recovery by caustic.

8. An NaOH concentration of 0.1M in the displacing liquid produces a maximum recovery efficiency.

9. The technique can be used to screen prospective operating

conditions and to investigate the effects of oil sand chemical composition on the recovery process.

ACKNOWLEDGEMENT

J.L. Margeson extends thanks to Energy, Mines and Resources Canada for funding this research under an agreement of cooperation with the University of Ottawa and also for permitting the research to be performed in the Energy Research Laboratories of CANMET.

REFERENCES

1. Farouq Ali, S.M. and Abad, B. "Bitumen recovery from oil sands using solvents in conjunction with steam"; J Can Pet Technol 15:3:80-90; 1976.

2. Singhal, A.K. "Physical model study of inverted seven-spot steamfloods in a pool containing conventional heavy oil"; J Can Pet Technol 19:3:123-134; 1980.

3. Redford, D.A. "The use of solvents and gases with steam in the recovery of bitumen from oil sands"; J Can Pet Technol 21:1:45-53; 1982.

4. Alberta Research Council OSRD Method 1.01; "Oil sand Dean Stark"; 1983.

5. Cragoe, C.S. "Changes in the viscosity of liquids with temperature, pressure and composition"; Proc World Petroleum Congress 2:529-541; 1933.

6. Takamura, K. and Chow, R.S. "A mechanism for initiation of bitumen displacement from oil sand"; J Can Pet Technol 22:6:22-30; 1983.

7. Hall, A.C., Collins, S.H. and Melrose, J.C. "Stability of aqueous wetting films in Athabasca tar sands"; Soc Pet Eng J 23:249-258; 1983.

Table 1. Analysis of medium grade Athabasca oil sand

Component	Wt %
Sand	83.4
Water	3.4
Bitumen	12.0
Losses	1.2

Table 3. Operating conditions

Variable	Value
Temperature, ^{o}C	45
Sand mass, g	40
Flowrate, cm^3/h	204
Pore volumes pumped	10

Table 4. Range of compositional variables

Variable	Lower level	Upper level
Sand drying temperature, ^{o}C	25	300
Sand size distribution	entire	-80+200 mesh
Added kaolin, wt%	0	3
Hexadecane dilution, wt%	10	20
Connate water content, wt%	1.0	3.4
NaCl in connate water, M	0	0.1
Displacing phase	water	0.1 M NaOH

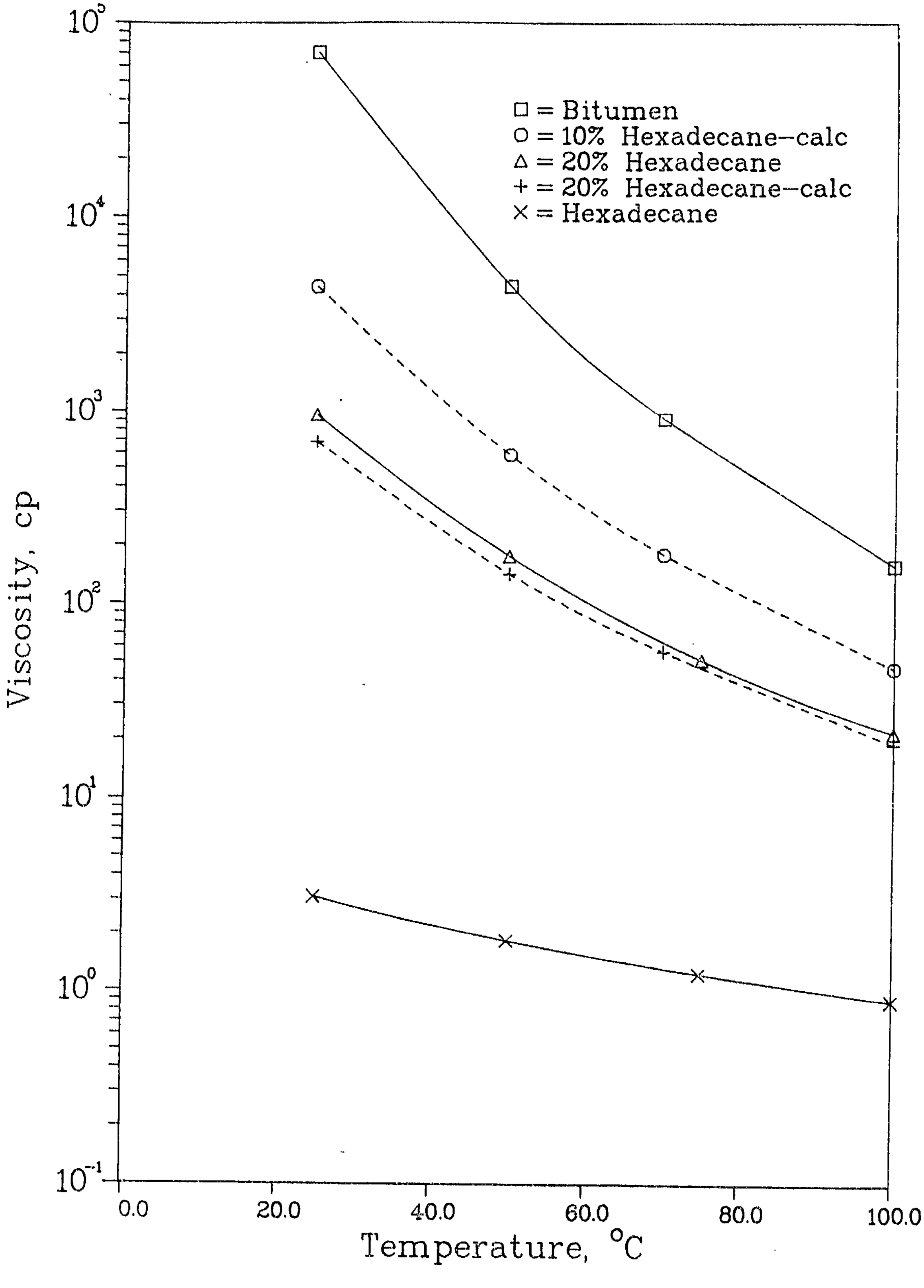

Figure 1: Comparison of calculated and measured mixed liquid viscosities.

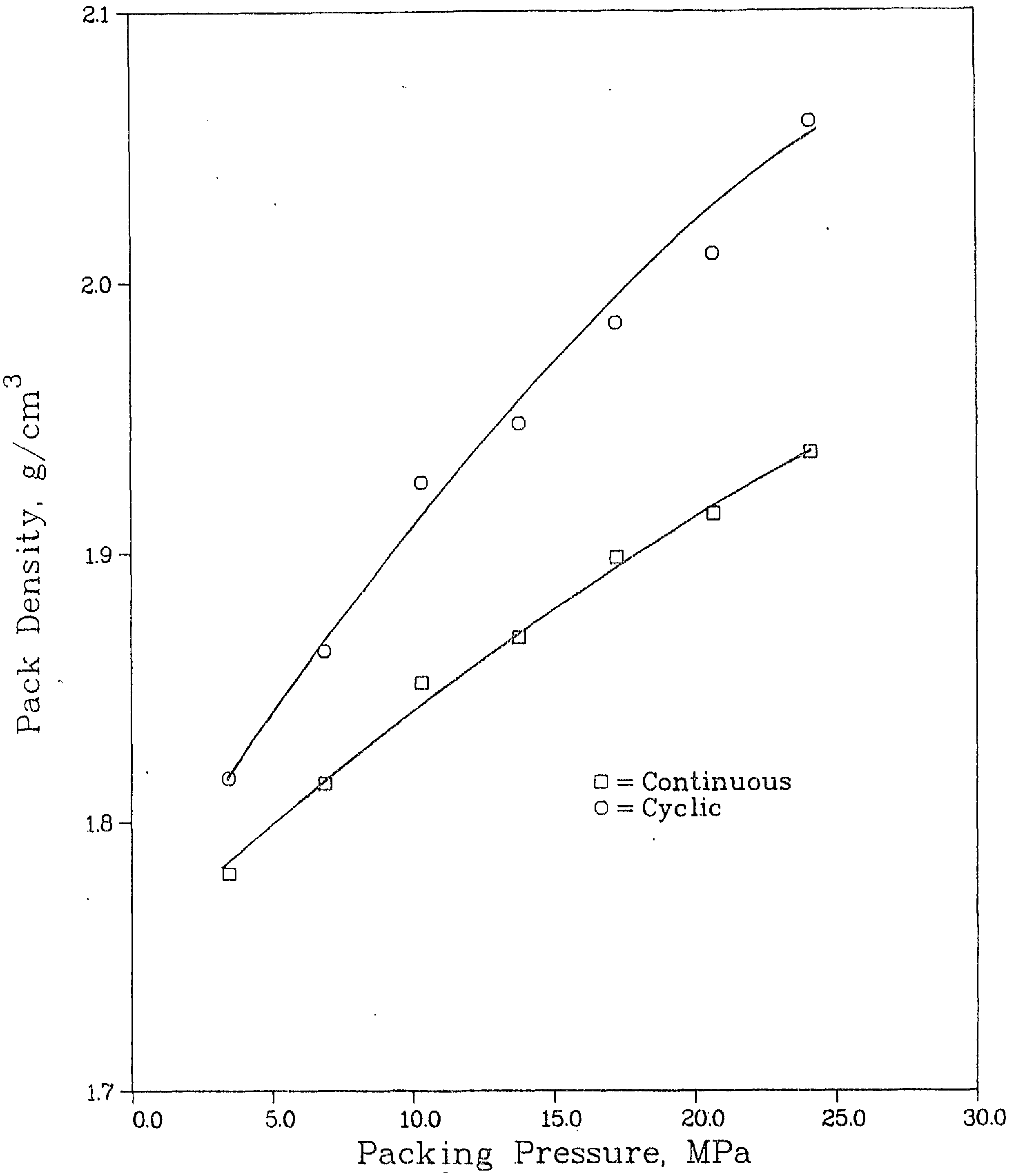

Figure 2: Influence of packing procedure on resultant oil sand density.

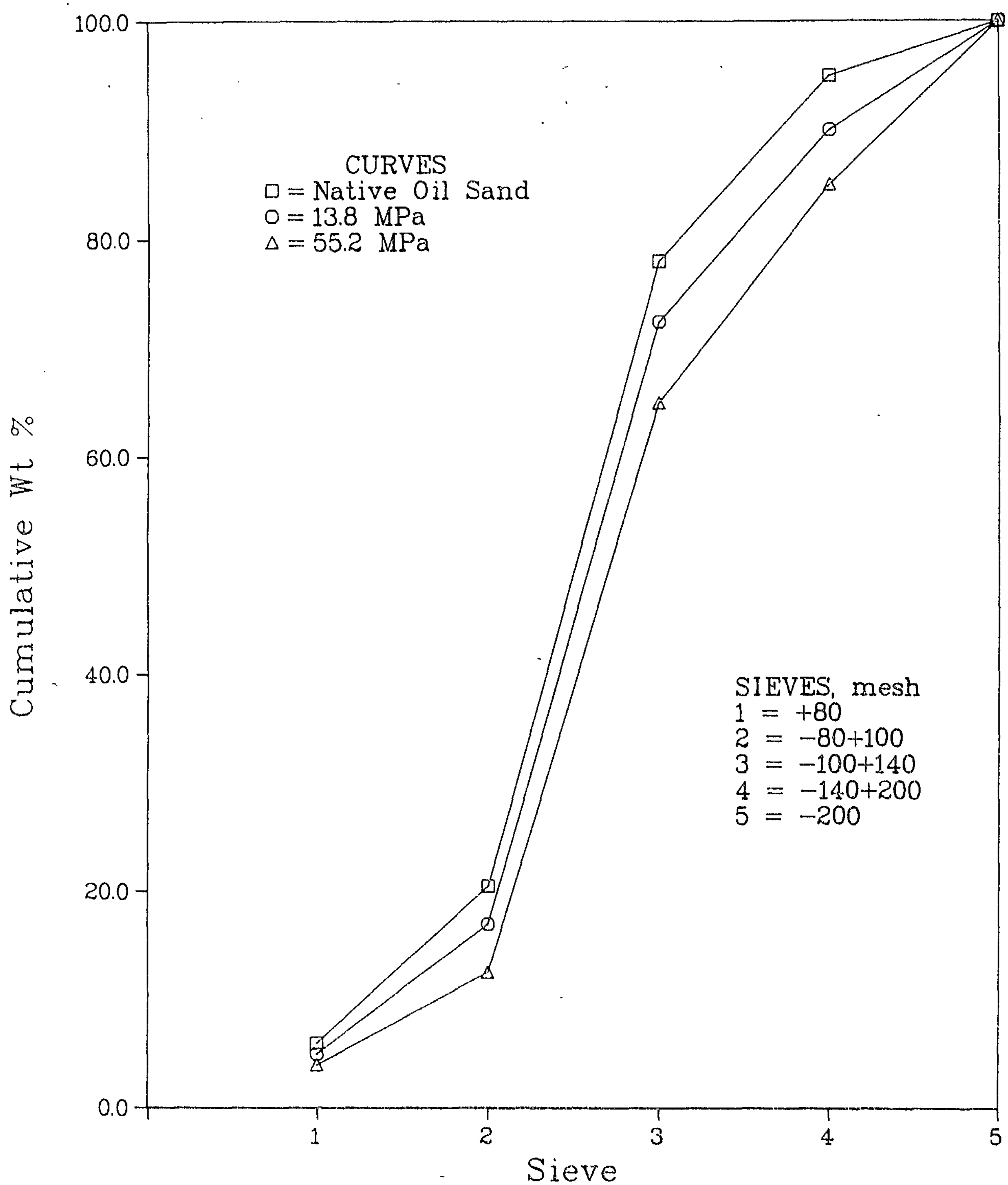

Figure 3: Sand size distribution related to compaction pressure.

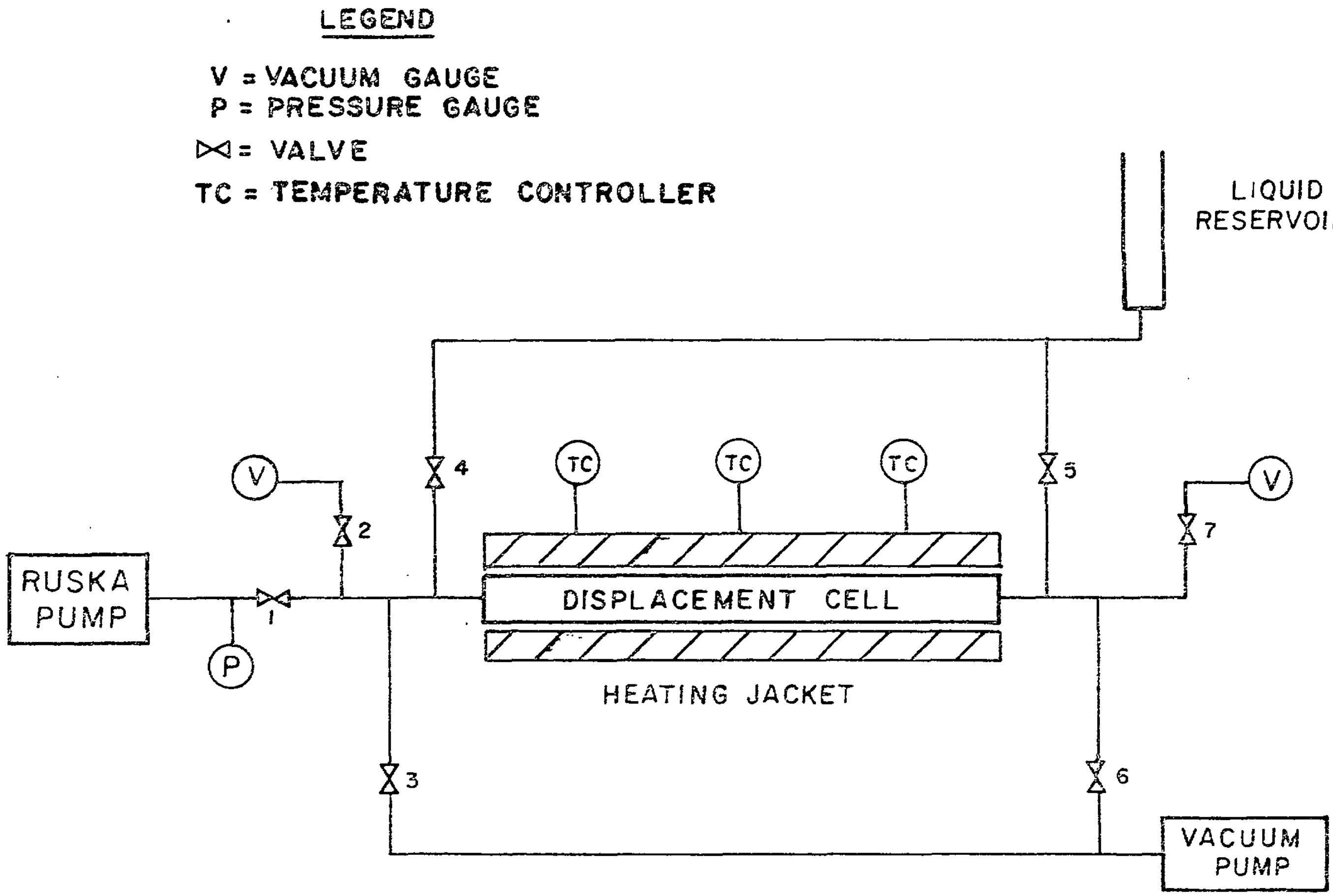

Figure 4: Experimental displacement apparatus.

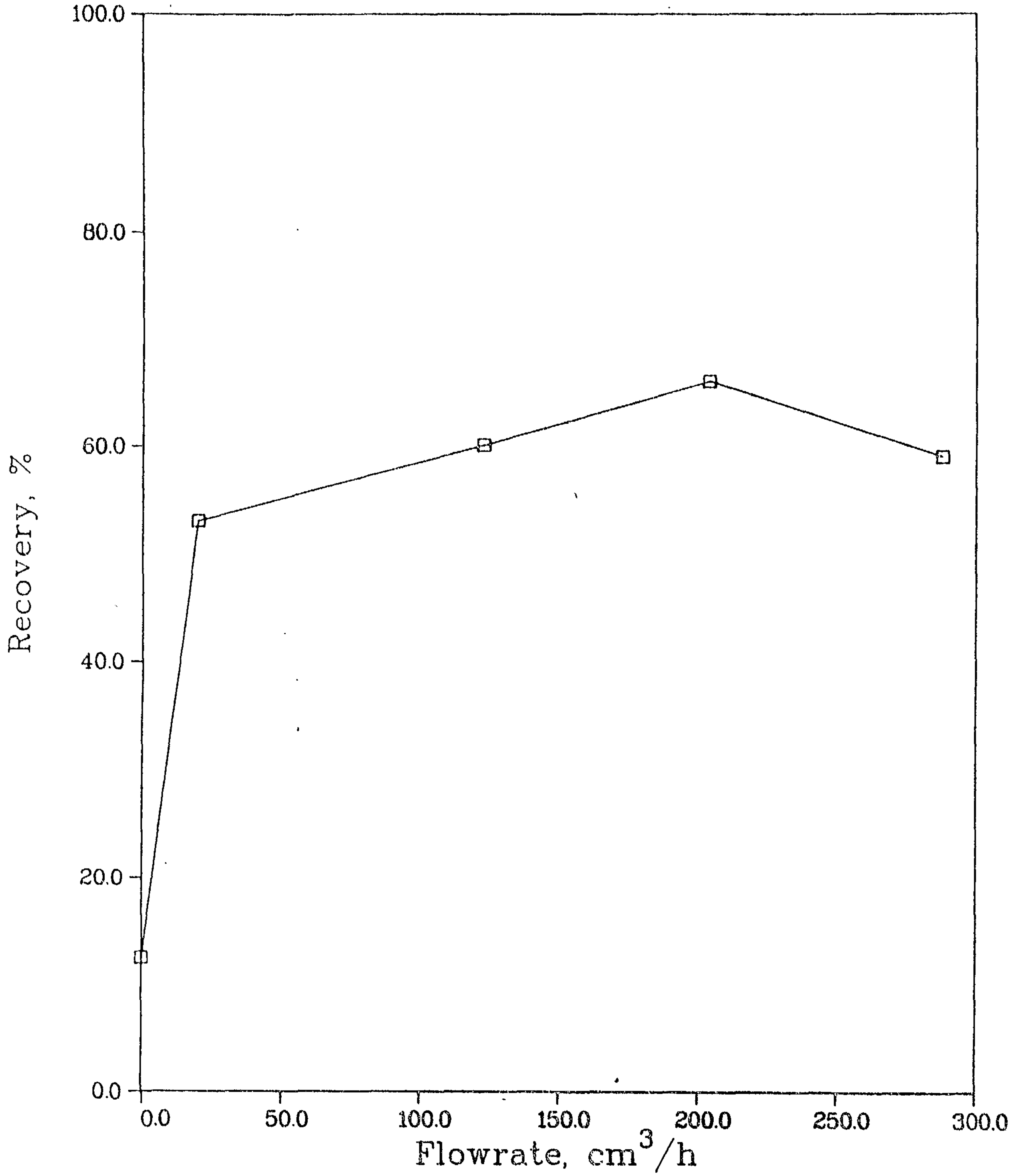

Figure 5: Influence of flowrate of caustic on displacement efficiency.

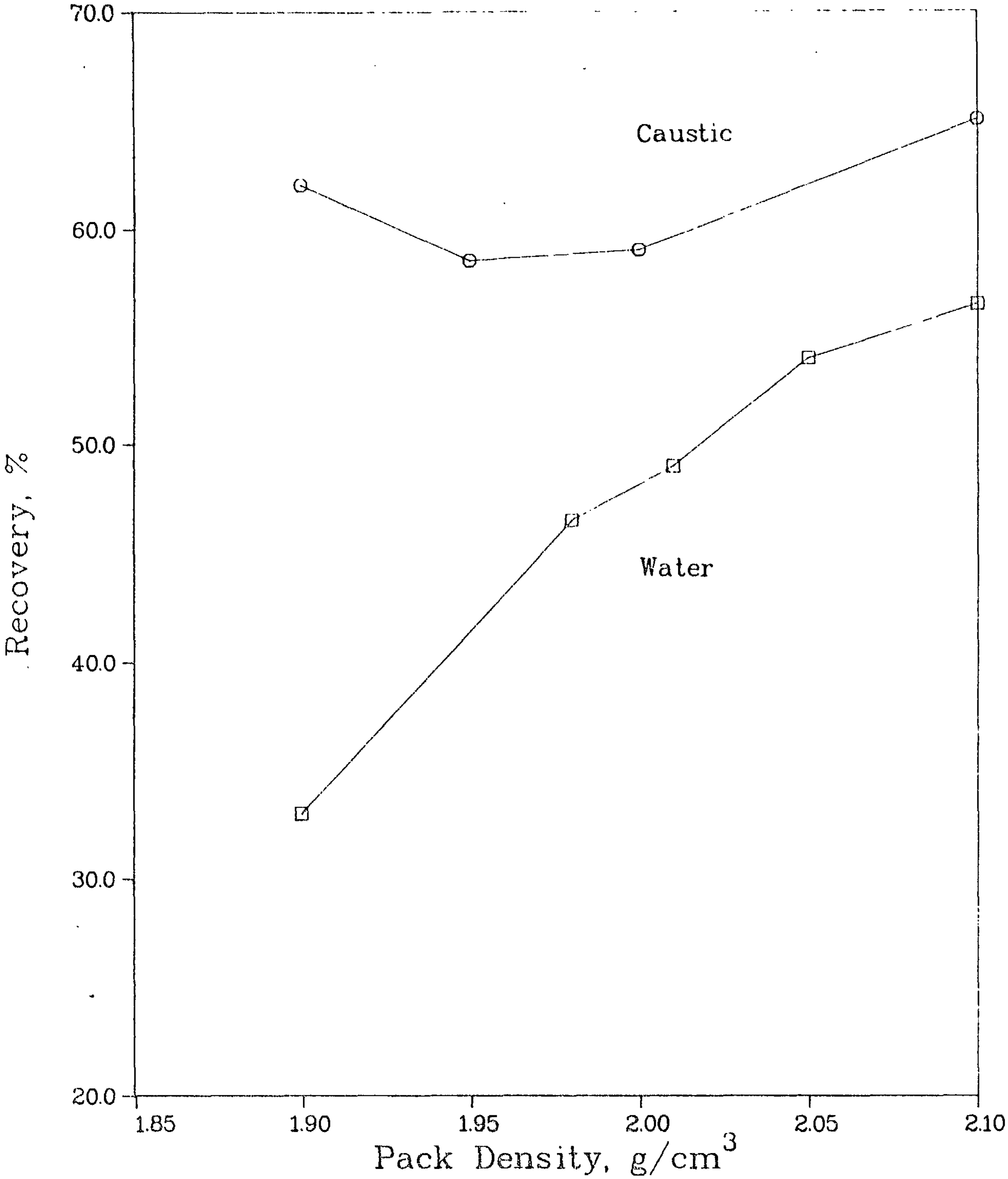

Figure 6: Influence of oil sand compaction density on recovery.

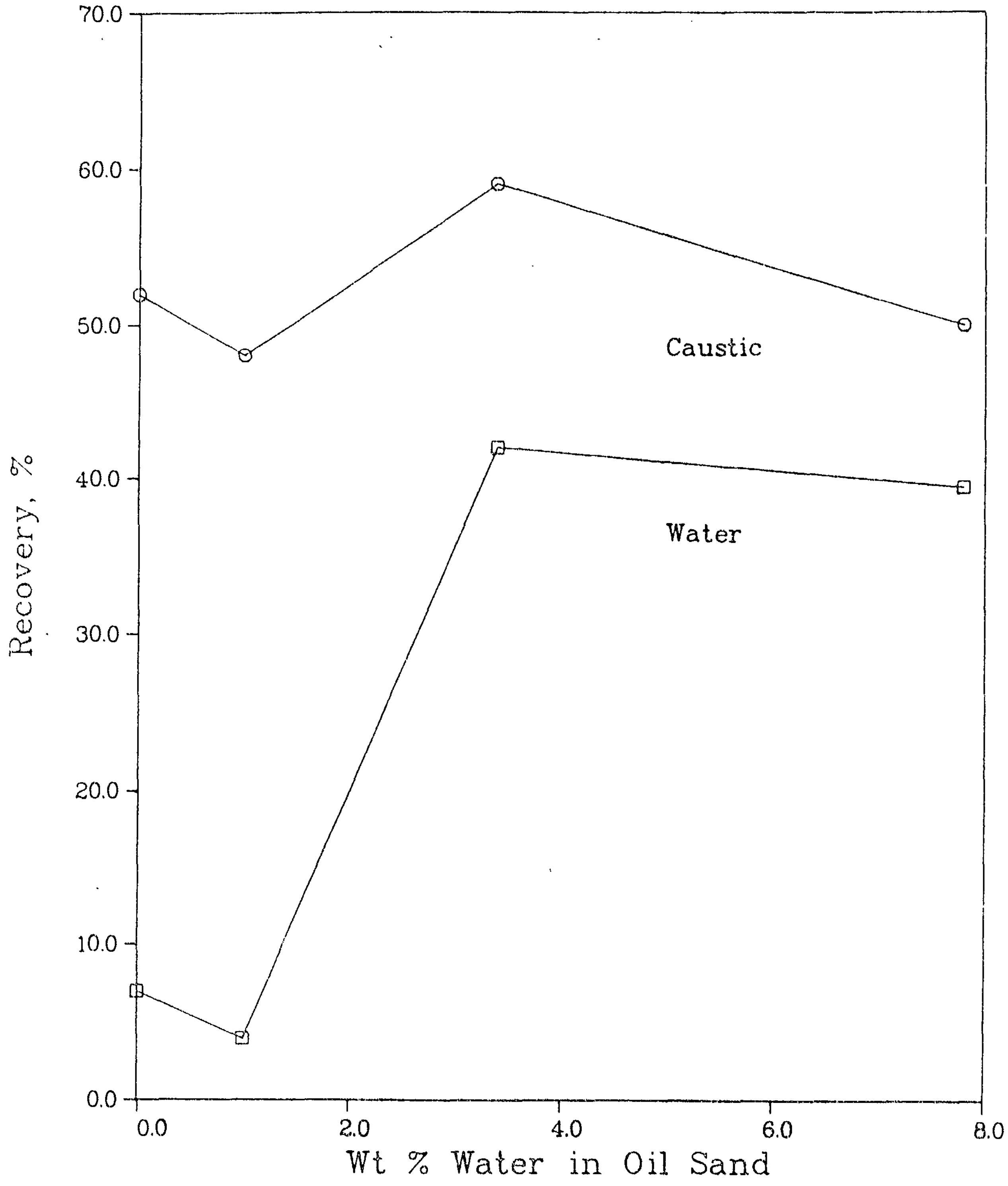

Figure 7: Effect of connate water saturation added during reconstitution on recovery.

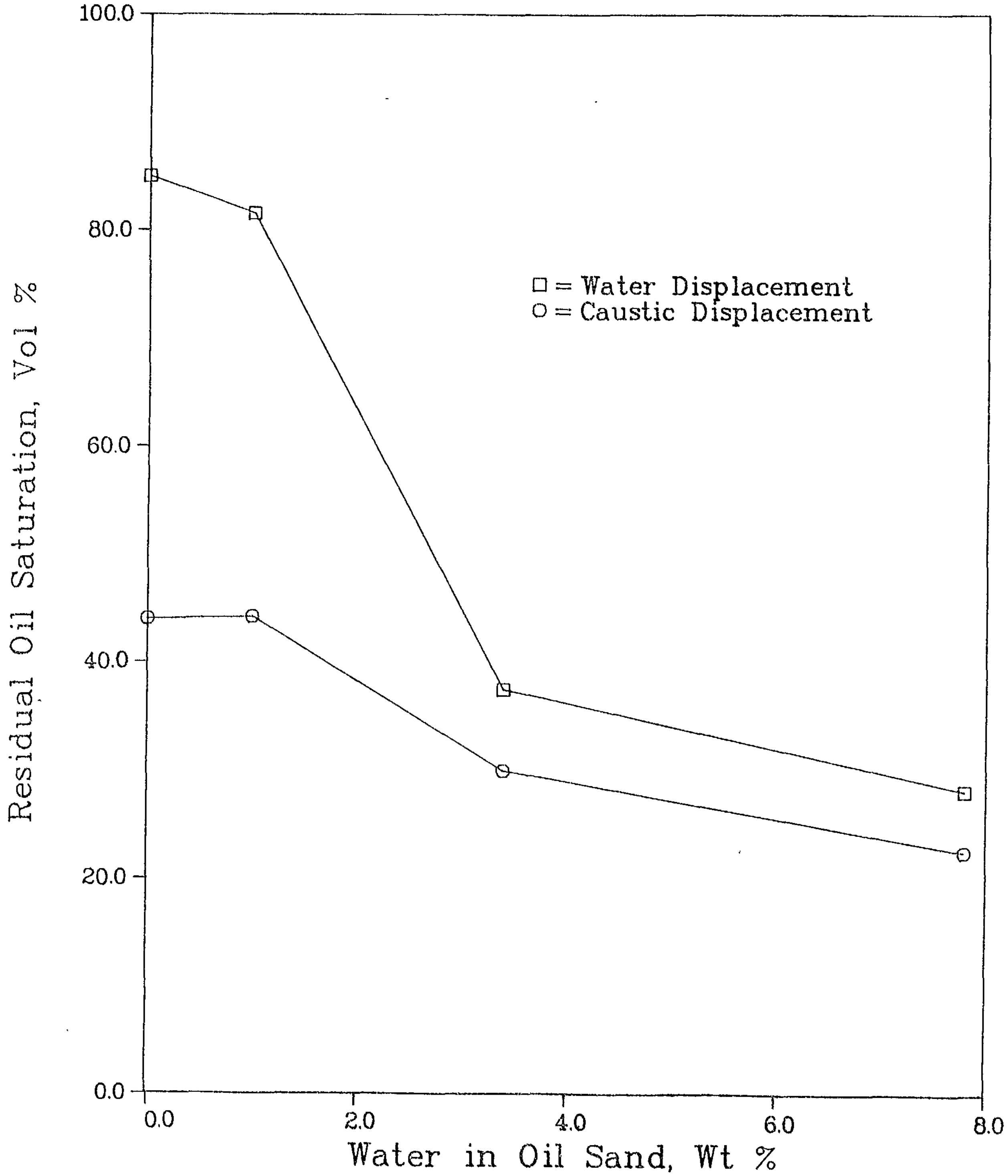

Figure 8: Effect of connate water saturation added during reconstitution on residual oil saturation.

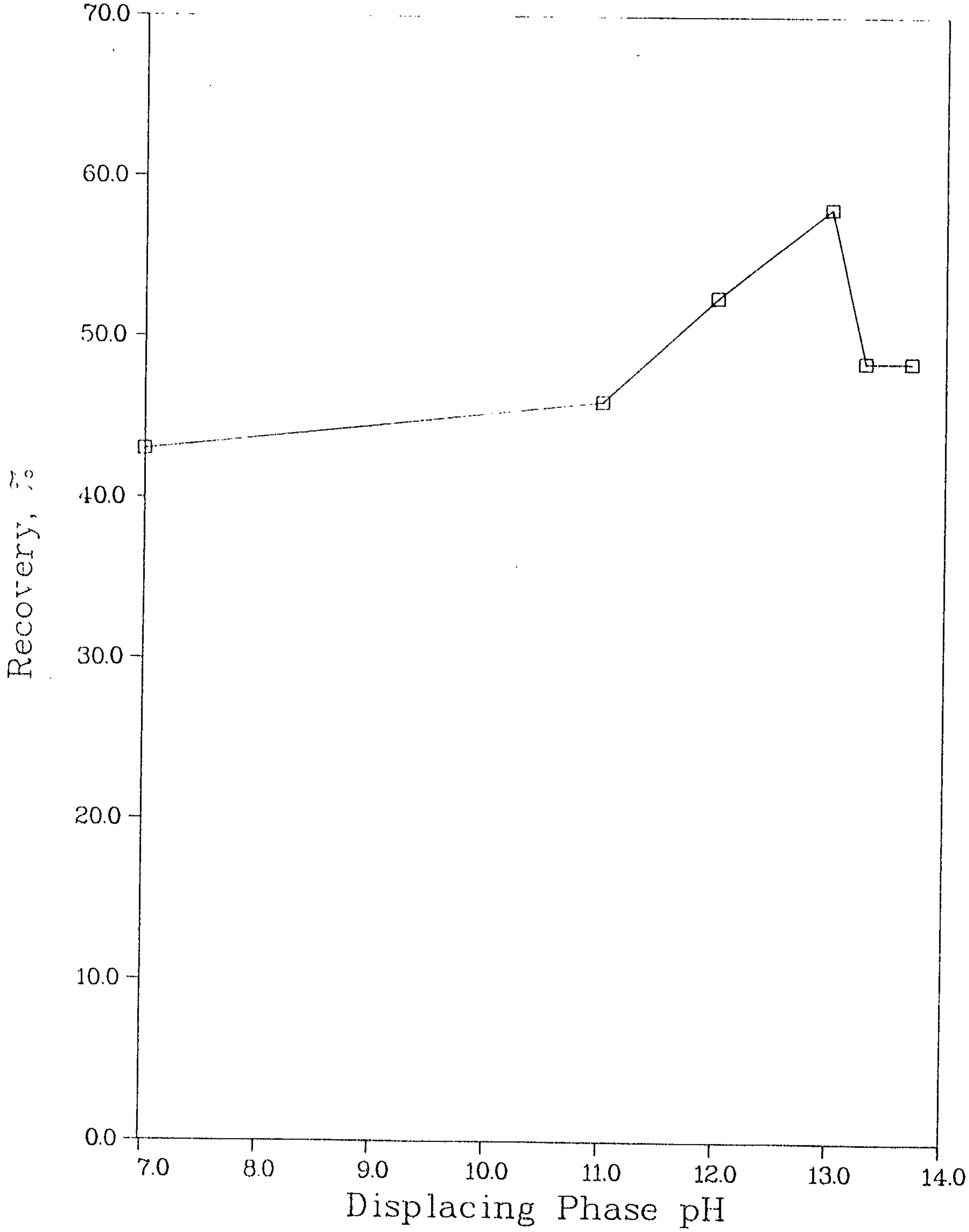

Figure 9: Effect of displacing phase pH on recovery.