THE FRACTIONATION OF CRUDE PETROLEUM BY CAPILLARY DIFFUSION. UNITED STATES GEOLOGICAL SURVEY

Published @ 2017 Trieste Publishing Pty Ltd

ISBN 9780649246656

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Edited by Trieste Publishing Pty Ltd.
Cover @ 2017

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Trieste

DEPARTMENT OF THE INTERIOR
UNITED STATES GEOLOGICAL SURVEY
GEORGE OTIS SMITH, DIRECTOR

BULLETIN 365

THE FRACTIONATION OF CRUDE PETROLEUM BY CAPILLARY DIFFUSION

BY

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UNDER THE SUPERVISION OF

DAVID T. DAY

WASHINGTON
GOVERNMENT PRINTING OFFICE
1908

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By J. Elliott Gilpin and Marshall P. Cram.

INTRODUCTION.

When, in process of refinement, black vaseline is filtered through warm, dry fuller's earth, the first product is an oil that is perfectly liquid at ordinary temperatures, but the succeeding portions are progressively more viscous until fairly hard vaseline is obtained. The observation that a fractional separation of oils in vaseline had been effected suggested to David T. Day that a like result might be obtained with crude petroleum. He applied this method to a sample of green-crude petroleum from the "third sand" of Venango County, Pa., and found that light products, chiefly gasoline, first appeared when such crude oil was allowed to filter down through a long glass tube filled with granulated or powdered fuller's earth.[a]

This result was followed by experiments with a more elaborate system of specially constructed funnels similar to those used by the refiners of vaseline in testing the comparative value of various fuller's earths. The results from these experiments were briefly summarized in a paper on the ability of petroleum to migrate in the earth.[b] Engler later verified these results and showed that the separation was mechanical and that no oxidation was effected in the process. Day next used a large closed funnel of galvanized iron holding about 100 pounds of fuller's earth. When crude petroleum was dropped slowly and regularly into this funnel, rather light oils were obtained at first, followed by the usual succession of heavier oils. As it was evident from this work that much of the oil passed through crevices without any change, Day tried the effect of reversing the route of the oil and of allowing it to diffuse upward through a tube packed tightly with fuller's earth. In such a tube the lighter constituents rose much more rapidly than the more viscous oils, so that by separat-

[a] Proc. Am. Philos. Soc., vol. 36, No. 154, 1897. [b] Trans. Petroleum Congress (Paris), 1900.

ing the fuller's earth from different sections of the tube and displacing the oil by water, very different oils were obtained from the upper and lower parts of the tube.

By using several tubes and uniting oils of the same specific gravity oil of different grades can be collected in sufficient quantity to be fractionated again, and the process can be continued until oils result which are not altered by further passage through tubes filled with fuller's earth. At the suggestion and with the cooperation of Doctor Day we have taken up this problem with the results here stated.

DETAILED DESCRIPTION OF EXPERIMENTS.

FRACTIONATION IN TUBES.

The tubes used first were of glass, 3 feet long and 1½ inches in diameter. They were closed at the lower end with corks along whose sides six or seven grooves had been cut, the inner end of the cork being covered with a bit of cotton cloth to prevent the earth from sifting out through the grooves. Such tubes filled with fuller's earth were placed with their lower ends in an open dish of petroleum and the oil was allowed to rise.

At room temperatures (18° to 22° C.) and atmospheric pressure, the rate of rise of crude petroleum in a tube filled with fuller's earth was very slow. In seven days the oil ascended but 73 centimeters in one tube and 59 centimeters in another, and in a third tube ten days were required for it to rise 59 centimeters. To study the effect of heat, a glass tube about 3 feet long and 1½ inches in diameter was filled with earth and placed in a bottle holding about 2 liters of oil, and the whole was heated by an electric stove with which temperatures considerably above those of the room could be maintained day and night. The temperature of the tube was kept between 40° and 70° for three days, in which time the oil rose 54.7 centimeters in the tube; in another tube packed in all ways like the first, but held at room temperature (about 20°), the oil rose 46 centimeters in the same length of time. With two tubes in which the earth was packed much less compactly the time required for the oil to rise 54 centimeters was four days for the tube at room temperature and two days for the one at 50° to 80°. The rate of rise was evidently but little affected by heat, at least within this range of temperature, and higher temperatures could not be used without loss of the more volatile constituents of the oil.

The next attempt at increasing the rate of rise of the oil consisted in applying diminished pressure to the top of the tube, which reduced the time required for the oil to reach the top of a tube 5 feet long from several weeks to seventeen hours. If diminished pressure is continued after the oil has reached the top and if the oil is not exhausted in the reservoir at the bottom, oil will be drawn over from the top of the tube. The specific gravity of the oil thus collected steadily rises as it comes over. The samples so obtained, however, stand under very low pressures for some time, which may cause a loss of their more volatile constituents. This suggested applying increased pressure to the oil in the reservoir rather than diminished pressure to the top of the tube, and an iron bomb, like those used for the transportation of mercury, was fitted with an iron pipe 7 feet long to contain earth and a side arm at the bottom of the bomb to which a water column might be attached.

The bomb, which held about 2 liters, could be partly filled with petroleum and the pipe containing the earth screwed into the top. The side arm which opened into the bottom of the bomb could then be connected with the water pressure so that the lower part of the bomb was filled with water which drove the petroleum upward. The oil obtained at the top, however, was fractionated no further nor in any larger amounts than when the oil was not allowed to emerge from the top of the tube. The difficulty of setting up such a pressure apparatus with tight connections, as well as the range of pressure required—a column of water 7 feet high being too great when the oil was just started up the tube and a column 30 feet high being insufficient when it was near the top—made its use impracticable.

To use diminished pressure, the earth in the tubes must not be packed so hard that the air just above the oil can not be drawn through the earth above, nor must the earth be packed so loosely that the oil will rise as in a vacuum. The right degree of hardness is obtained by filling about 1 foot of the tube at a time and packing that much earth as hard as possible with a wooden rod tipped with a rubber stopper. If the tube when pounded on the floor rings in the hand, it indicates that the earth may be packed too closely. Tubes may be packed much more easily by filling several at once, with a separate ramrod for each. By allowing a few minutes to elapse between successive liftings of the ramrod, much labor is avoided. A bit of cotton waste below a rubber stopper at the top of the tube will prevent any earth from being drawn up when the air is exhausted.

The fuller's earth was first heated in shallow iron pans until it ceased to form geysers when stirred. The earth must be thoroughly cold before it is packed into tubes, or the contraction will be sufficient to allow the oil to run up the tube immediately when the air is exhausted.

Much trouble was experienced with the tubes first used on account of their breaking—not when in service, but soon afterward. This was thought to be due to the age of the tubing, but the same thing hap-