5-2

Memo. Series No. 9

AN INVESTIGATION OF THE ECONOMIC VALUE OF A FOSSIL RESIN FROM BRITISH COLUMBIA*

By R. T. Elworthy and R. K. Carnochan.

Recently the presence of a fossil resin in the coal on waste dumps at the Coalmont Collieries, Coalmont, B.C., aroused some interest especially as it was reported to be identical with Prussian amber. The following accounts of the chemical and physical properties of the resin, its possible industrial applications and the most suitable methods of separating it from the coal in which it occurs are the results of work that has been carried out in the laboratories of the Mines Branch.

CHEMICAL AND PHYSICAL PROPERTIES AND POSSIBLE INDUSTRIAL APPLICATIONS.

R. T. Elworthy, B.Sc., F.C.I.C.

Fossilized resinous substances have been known to exist in certain regions in the west since the earliest explorations of prospectors and geologists. Dr. Harrington in the Geological Survey Report for 1876-77 recorded the results of examination of three samples, one from the North Saskatchewan river, one from Peace river, and another from Nechacco river, south of Fort Fraser, B.C.

Lately some interest has been aroused by the presence of a fossil resin in coal from the Coalmont Colliery, Coalmont, B.C., especially because the substance was reported to be amber.

The following paper is a brief summary of work that has been carried out on this fossil resin in the laboratories of the Mines Branch, particularly stressing its comparison with amber and its possible industrial uses.

* Prepared for presentation at the Annual Western Meeting of the Canadian Institute of Mining and Metallurgy, November 1922. By permission of the Director, Mines Branch, Department of Mines, Ottawa.

Amber.

Amber is the specific name given to a hard fossil resin, varying in colour from lemon yellow to deep brown, sometimes transparent but usually translucent or opaque, chiefly found on the eastern shores of the Baltic where it is mined under the monoply of the German Government. The mineral occurs in nodules disseminated in a blue clay of cretaceous formation or associated with lignite in clay and sand, sometimes under the sea. Thousands of pounds were produced annually before the war.

Amber is used chiefly for ornaments, pipe stems, and cigarette holders. Years ago it was highly valued for varnish making, but it has since been displaced by the cheaper eastern copals.

The main characteristics that differentiate amber from other fossil resins are (i) its greater hardness, (ii) the presence of succinic acid in the products of distillation.

Several varieties of amber are known, such as succinite, retinite, gedanite, and glessite; sometimes the term amber is restricted to the variety succinite.

The Coalmont fossil resin.

This substance is found in the lower grade coal, close to the slate contact of the coal seams and was first observed in the coal of the waste dumps.

A lot of 400 pounds of this coal was received by the Department. The resin formed about two per cent of it. The pieces, about the size of peas, were irregular in shape with no marked cleavage lines and were fairly brittle. They varied in colour from opalescent lemon yellow through all shades of brown to black. Some pieces showed a green fluorescence. As described in another section the resin was easily separated from the coal by flotation in solutions of sodium or calcium chlorides of specific gravity 1.10.

This material was further subjected to a grading process, using solutions of specific gravity 1.03, 1.05, and 1.09 giving four fractions of marked differences of colour.

For many of the tests, samples of pure resin were hand picked.

In the following paragraphs, the values for the chief physical and chemical constants are briefly recorded. Comparative experiments on the Coalmont resin and on authentic samples of Prussian amber were usually carried out.

<u>Specific gravity.</u>

The results varied from 1.031 to 1.168. This variation in density, common to most fossil resins, is due chiefly to occluded gas bubbles. The specific gravity of Prussian amber is given by Dana as 1.060 to 1.096.

<u>Hardness.</u>

Most pieces scratched gypsum (hardness 2) but not calcite (hardness 3). The hardness of amber is recorded as 2.5.

<u>Softening and Melting Points.</u>

Prussian amber begins to soften at about 150°C at atmospheric pressure, becoming completely molten between 280°C and 300°C. The coalmont resin behaved very similarly, softening between 160° and 180°C and melting between 270°C and 300°C.

<u>Products of distillation.</u>

When amber is distilled, the initial products are water and a light lemon-coloured oil. As the temperature rises the oily distillate is darker in colour. The vapours have a characteristic smell. Coalmont resin on distillation gave a much less watery distillate, but the oil closely resembled amber oil. The quantity of oil obtained was about 70 per cent of the resin. The specific gravity of the oil was 0.939 at 20°C. It was soluble in ether, chloroform, and carbon tetrachloride but only partially soluble in absolute alcohol.

On redistillation three fractions, collected between 140-180°C., 280-310°C., and 350-370°C., were obtained, the first yellow, the second pale green, and the third brown in colour. All the distillates darkened on standing, especially on exposure to air. A dark brown resinous substance forming 20 per cent of the original oil remained. It was soluble in gasoline and in turpentine.

<u>The Succinic acid content of the resin.</u>

A sample of 50 grams of amber was heated in a distillation flask and the products of reaction collected. The water was separated from the oil by filtration and on evaporating, a mass of crystals remained. These were purified by recrystallisation from absolute alcohol and weighed 2.1 gm., equal to 4.2 per cent of the amber. Tests proved the material to be succinic acid.

Several samples of the Coalmont resin were treated similarly. The aqueous portion of the distillate had an acid reaction and gave faint indications of succinic acid but on evaporating only a resinous material remained, there not being sufficient succinic acid to determine quantitatively.

The action of solvents.

The solubility of the Coalmont resin in alcohol, ether, acetone, chloroform, carbon tetrachloride, pyridine, selenium oxychloride and similar solvents was slight, never more than 40 per cent of the resin dissolving.

It was soluble in linseed oil and turpentine after being heated to 300°C, as amber is.

Analytical constants.

The saponification value, acid value and iodine value were found to be of the same order as similar figures for Prussian amber. Ultimate analyses gave a composition of about 80 per cent carbon, 10 per cent hydrogen, 9 per cent oxygen, 0.3 per cent sulphur and 0.7 per cent ash.

Conclusions.

This summary shows that the Coalmont resin closely resembles most variaties of Prussian amber in its physical and chemical properties though it would not be considered true amber or succinite from a mineralogical point of view.

The possible Industrial Uses of Coalmont Resin.

Varnish Manufacture.

Several samples of varnish were made up in the laboratory according to prescribed recipes by melting the resin, adding hot boiled linseed oil and small quantities of litharge as a drier, and thinning with spirits of turpentine. After filtering and leaving for some time these were tested and compared with some typical commercial varnishes. They gave very satisfactory results, drying in the usual time, and resulting in lustrous and hard surfaces. The chief disadvantage was the rather dark colour. Samples of the graded resin have been submitted to various varnish manufacturers though unfortunately not in sufficient quantity for plant tests to be made. Their opinions have been without exception, that, although the resin would undoubtedly make a good varnish, it could not compete with the large sized and much paler copals such as Zanzibar, Kauri, Manila and Congo gums. One has only to see these resins contrasted with the Coalmont resin to be equally convinced. The disadvantages are its small size and its comparatively dark colour, especially evident when it is heated.

The greatest demand is for pale varnishes and the highest price is obtained for large sized, pale and clear resins. For the cheaper and dark varnishes Kauri scrapings and dust can be obtained for a few cents per pound and much rosin is also used. It is only with these materials that the Coalmont resin could compete and even then it would be difficult to convince the varnish makers that the harder Coalmont resin is superior.

The oil obtained by distillation.

There is very little use for amber oil. Similarly it would be difficult to find uses for the oil obtained by distillation of the Coalmont resin. Its property of darkening on exposure to light and air make it unsuitable for most purposes. It might be used to add to cements, artificial resins, and plastics but its hardening and water-proofing properties are doubtful.

Amber oil is quoted at 75¢ to $1.00 a pound on the New York market. Coalmont resin oil is very similar in its properties to amber oil.

Moulding processes.

Large sized blocks of amber are formed from chippings and waste by the action of heat and pressure. According to one process the material is heated to 200-250°C and 400 atmospheres pressure is applied. Experiments made compressing Coalmont resin have not yet been successful though it is hoped to obtain better results with a new mould now being made in the workshops.

But the synthetic resins such as Bakelite, Redmanol and Condensite are rapidly supplanting compressed amber and it is improbable that there is any use for the Coalmont resin in this way.

It, therefore, seems difficult to find satisfactory industrial uses for the resin unless it can be obtained in considerable quantity and sold cheaply enough to compete with the darker varnish resins.

SEPARATION OF FOSSIL RESIN FROM COAL

By R. K. Carnochan, B.Sc.

SHIPMENT: 450 pounds of coal containing fossil resin was received at the Ore Dressing and Metallurgical Laboratories, from the Coalmont Collieries, Coalmont, British Columbia.

CHARACTERISTICS: On examination of the shipment it was found that the coal was an impure variety of bituminous, containing a considerable quantity of slate. Fossil resin up to one half inch in thickness was found associated, the greater proportion, however, being considerably thinner - less than one quarter inch - showing that there was little possibility of obtaining pieces that could be used for the manufacture of pipe stems, ornaments, etc. In fact, the coal had to be crushed to four mesh before any appreciable amount of resin was freed.

METHOD OF SEPARATION: Flotation of the resin from the coal in an aqueous solution of sodium chloride or some other salt. The resin floats on the surface, and the coal sinks in the same solution.

PROCEDURE FOLLOWED IN CONDUCTING TESTS: Six tests were made on the shipment, five on a small laboratory scale on about 20 pounds of material, and one on a larger scale on 300 pounds. The procedure in all was practically the same, with the exception that calcium chloride solution was used in one case instead of sodium chloride, and in the larger test, the coal was ground to 20 mesh before separation. In the tests in which the coal was crushed to four mesh, it was found that while a considerable quantity of the resin was freed and could be floated at this mesh, it was necessary to grind the tailing to 20 mesh and refloat, to obtain a clean tailing.

After crushing the coal to the necessary fineness, the material was fed into the centre of an agitator containing a solution of sodium chloride, or calcium chloride, density 1.15 to 1.17, with a spray of solution to wet the fine particles of coal and cause them to sink in the solution. The coal was drawn out from the bottom of the cone agitator in a thickened pulp, and the resin floated to the surface, flowing off into the circular launder to the main launder over a 100 mesh screen, to

58

remove the aqueous solution which was used over again. The resin was washed, dried, and weighed.

<u>RESULTS OF TESTS:</u> The resin recovered in the above manner from the coal represented about two per cent of the original feed. A close inspection of the products under the microscope showed the resin to be free from coal, and the coal tailing to be clean, denoting a high recovery of the resin.

A solution of sodium chloride gives just as good results as calcium chloride, and is cheaper to use although more is required for the same density of solution.

In the separation of the resin from the coal as represented by this particular sample, in which the resin was not entirely freed from the coal until crushed to 20 mesh, crushing to this mesh is advisable as it permits of a more simple operation, and does not require regrinding and a double separation.

The separation is simple and quite feasible for practical operation on a large scale, provided that there is sufficient resin in the coal, and that an economic market could be obtained for such a product.

The results of the test work will be given in detail in the Summary Report of the Mines Branch for 1922.

———— o ————