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EXHAUSTIVE EXTRACTIONS OF A MINNESOTA PEAT

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In 1937 a group of Irish workers¹ carried out a comprehensive study of the extraction of waxes from an Irish peat by various pure solvents and solvent mixtures. In this work they used only such solvents as can form azeotropes with each other, and each mixture had the azeotropic composition. (This decision was based on the fact that under continuous Soxhlet-extraction conditions, only an azeotrope will have constant composition, both of the liquid and vapor, and thus be capable of affording reproducible results. In fact, in many non-azeotropic mixtures whose components differ substantially in their boiling points, there is sufficient fractionation that only the more volatile member can even reach the extraction thimble, so that an extraction by such a "mixture" is really nothing more than an extraction by a single solvent.) Most of the common solvents then commercially available were used in this research. The yield of waxes varied from

¹ J. Reilly, D. F. Kelly, and D. J. Ryan, J. Soc. Chem. Ind. 56, 231T (1937).

5.2 to 18.6 per cent, depending on the solvent or solvent mixture. In the opinion of these workers, the extracts seemed to have commercial possibilities, both as waxes and as sources of fatty acids and other high-molecular-weight compounds.

Since then various other investigators have examined the extraction of waxes from peat, but for the most part using the same solvents or solvent mixtures as the Irish group. Sundgren and his co-workers, for example, have done considerable work on the extraction of waxes from Finnish peats. At the recent Dublin symposium² they reported the extraction of waxes from Finnish and other peats with benzene-ethanol or with trichloroethylene-ethanol, with yields about 10 per cent on a dry basis. Describing their production of a stabilized peat wax which they call "S.E.," they were quite optimistic about the commercial possibilities of this product, both as a wax and as a starting material for transformation to useful chemical derivatives. At the same conference, Ackroyd³ described recent British work on solvent extraction of waxes from British peats.

Since the early work of Reilly, Kelly, and Ryan,¹ numerous solvents which were formerly known only as rare and costly laboratory-scale chemicals have become commercially available on a large scale. A number of these have unusually strong solvent powers and have become particularly valuable in several industries, such as plastics and synthetic rubbers; but since these solvents are relatively new, their action on peat has not been studied to any appreciable extent. We therefore felt that it would be profitable systematically to study the action of these solvents and their azeotropes on Minnesota peats. This research is now well under way, and this report describes the progress to date.

The peat used in these experiments was taken from the Rice Lake bog—about 10 miles north of Duluth. The sample was air-dried, ground in a feed mill, then further dried for at least 24 hours at 110° C. in a vacuum oven at about 50 mm. The anhydrous sample was weighed in a fritted-glass Soxhlet-extraction thimble, and subjected to continuous Soxhlet extraction at atmospheric pressure by the particular pure solvent or azeotropic mixture until the downsiphoning liquid no longer was colored, plus 6 additional hours. In each extraction the system was protected against atmospheric moisture by either a calcium chloride or soda-lime tube. Each solvent was commercial grade, and anhydrous (e.g., absolute ethanol rather than 95 per cent, 98 + per cent formic acid rather than 90 per cent, etc.)

At the end of the extraction period the residue in the thimble was allowed to drain, and air-dry if possible, then dried in the vacuum oven under the same conditions as the original sample, and weighed. The per cent loss in weight was then taken as the fraction extracted.

These extractions, which will be more fully reported elsewhere, have now been completed with 21 pure solvents and 10 binary azeotropes of these. The choices of solvents were based on several factors: A

² A. Sundgren, E. Ekman, and V. T. Rauhala, in section E. 1 of "Papers Presented at the International Peat Symposium," Bord na Mona, Dublin, 1954.

³ G. C. Ackroyd, *ibid.*

number of amines were used because of the probability that they would prove effective solvents for the humic acids in peat; dimethylformamide and several Cellosolves because they are relatively new in technology and useful in numerous other applications; benzene and ethanol because they are standard solvents for peat wax; others because they form azeotropes with the above.

Extraction by the pure solvents ranged from 2 per cent (with chloroform, in 13 hours) to 55 per cent (with ethylenediamine, in 140 hours), and by the azeotropes, from a 5 per cent *gain* in weight (by acetone-diethylamine, in 12 hours) to 56 per cent extraction (by 2-butanone-*n*-butylamine, in 202 hours).

An obvious and reasonable prediction as to the effect of boiling point of solvent on per cent extraction would be that the one should increase with the other. Such a temperature-solubility correlation, for any given solute, frequently holds regardless of functional-group variations⁴ and is almost inevitably true when the solvents are all members of the same homologous series. No such correlation was found in this work with peat, not even with several pairs of solvents of the same homologous series. Thus we find formic acid (b.p. 100°, extraction 34 per cent) and acetic acid (b.p. 118°, extraction 6 per cent), acetone (b.p. 56°, extraction 11 per cent) and 2-butanone (b.p. 79°, extraction 4 per cent), and similar situations with several other such pairs. Whether or not this is significant remains to be seen, by extending the extractions to include several more members of each of these homologous series.

In comparing the extractions by the azeotropes with those by the corresponding single solvents, several types of behavior may be anticipated: (a) Where each of the two solvents extracts essentially the same components, the azeotrope should extract approximately the same fraction of the peat as the stronger of the two solvents. (b) Where each of the two solvents is selective in its extraction, then the azeotrope may extract some fraction greater than that by the stronger solvent, up to the sum of the individual extractions. (c) Another possibility, one that is quite interesting but not entirely unexpected, is for the azeotrope to extract *more than the sum* of the individual extractions. This reflects some property of the combination of solvents not characteristic of either solvent alone and is one of the chief reasons for using an azeotrope. (d) the fourth mode of behavior of an azeotrope is the extraction of *less* material than by the better solvent alone, due, possibly, to chemical interaction of the solvent with the material being extracted. In the 10 extractions by azeotropes, examples were found of each of these four types of behavior.

ACKNOWLEDGMENT

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⁴ For example, in the extraction of coal by various aromatic solvents. See C. Golumbic, J. B. Anderson, M. Orchin, and H. H. Storch, U. S. Bureau of Mines, Report of Investigations 4662 (1950); M. Orchin, C. Golumbic, J. E. Anderson, and H. H. Storch, U. S. Bureau of Mines, Bulletin 505 (1951).