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A Study of Montana Bentonite

William P. Ryan

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A STUDY OF
MONTANA BENTONITE

by
WILLIAM P. RYAN

A Thesis
Submitted to the Department of Geology
in Partial Fulfillment of the
Requirements for the Degree of
Bachelor of Science in Geological Engineering

MONTANA SCHOOL OF MINES
BUTTE, MONTANA

May 29, 1933

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A STUDY OF MONTANA BENTONITE

Introduction

Research has been undertaken both under Government and private auspices in an endeavor to develop uses for bentonite. Perhaps, the work done to date has had only in consideration the possible industrial importance of bentonite. No simple, quick methods for the determination of the properties or qualities of any particular bentonite have been developed.

The work contained in this paper was carried on under the direction of the geologic department of the Montana School of Mines in an attempt to establish whether or not there is a means of making rapid simple determinations of the quality of Montana bentonitic clays, and in particular, with regard to the uses to which the clays may be suited. The problem also involves a study of Montana bentonite, and a comparison of it with the standard accepted bentonites and fullers earth on the market today.

The writer desires to acknowledge, with much appreciation, the direction and assistance given him by Dr. E. S. Perry and Dr. A. E. Koenig in carrying out the laboratory work necessary in the preparation of this report. The bibliography appended will enable those interested to refer in greater detail to the sources of information used in the preparation of this paper.

History of Bentonite

The first shipments for commercial purposes were made in 1888 by William Taylor of Rock Creek, Wyoming, after whom the material was called "taylorite." Ten years later it was

learned that the name taylorite had been used in mineralogic nomenclature, so the new material was designated as bentonite, because of its occurrence in the Fort Benton formation of the Rock Creek district.

At the time of original discovery, considerable interest was aroused in the industrial possibilities of bentonite, though slight success was experienced in utilizing the material and little has been heard of it until recently. This lack of success probably accounts for the fact that very little information regarding bentonite has been published except brief mention in Government reports and technical journals.

Occurrence

Bentonite usually occurs in beds from a few inches to many feet in thickness, mainly in Cretaceous sediments, but to some extent in the Paleozoic and Tertiary rocks in many parts of the United States and Canada. Ross¹, Shannon², and others have shown that most bentonite deposits are the result of devitrification and partial decomposition of glassy volcanic ash. Their conclusions were based largely on thin sections of bentonite with the characteristic structures of volcanic ash, the presence of feldspar, an absence of an appreciable quantity of quartz, the chemical composition, field relationships, and the widespread distribution of individual beds, often very thin and of fairly uniform thickness over great distances, such as would result from the settling of ash from a volcanic eruption.

Although most bentonites are believed to have been formed from volcanic ash, Ross and Shannon have pointed out that some

deposits have been formed in situ by the devitrification not only of glassy igneous ash or tuff but occasionally of lava flows and even of hypabyssal intrusives.

Bentonite outcrops are unique and striking. Since little vegetation will grow on them, they are barren, and because of the peculiar physical properties of bentonite, these weathered outcrops often present a crinkled or coral-like surface. This effect has been studied by Twenhofel, who concludes that the surface cracks are caused by the lag in absorption of water by bentonite.

Bentonite has been mined and produced in Wyoming, California, Nevada, South Dakota, Alabama, Kentucky, and Tennessee. Other states in which bentonite is known to exist are: Montana, Idaho, Utah, Oregon, Washington, New Mexico, Oklahoma, Kansas, Wisconsin, Louisiana, and Texas.

Fullers earth, which has properties quite similar to bentonite, is found in sedimentary rocks of lower Mesozoic up to Pleistocene in a number of the states, including Florida, Georgia, Alabama, Arkansas, Colorado and South Carolina.

Properties

Bentonite is composed of a group or series of clay-like minerals characterized by the presence of an alkaline oxide, an alkaline earth content of five to ten per cent, small grain size, high adsorptive power, and usually very strong colloidal properties.

Bentonite contains seventy-five per cent or more of the crystalline clay-like minerals, montmorillonite or beidellite, which are sometimes formed when igneous material decomposes. Montmorillonite is designated as $(\text{Mg, Na, Ca})\text{O} \cdot \text{Al}_2\text{O}_3 \cdot 5\text{SiO}_2 \cdot n\text{H}_2\text{O}$, with n equal to about 8. Larsen and Wherry³ used the name beidellite and assigned to it the formula $\text{Al}_2\text{O}_3 \cdot 3\text{SiO}_2 \cdot x\text{H}_2\text{O}$, in which x frequently is equal to 4, and the alumina is replaceable by other oxides. Varying amounts of other minerals may be included as impurities.

According to C. S. Ross⁴, recent microscopic and X-ray studies on bentonite indicate that it possesses crystalline rather than colloid character, and that the material probably consists, in the main, of the mineral leverrierite. He considered the pseudo-colloidal behavior of the clay in aqueous suspensions due to the micaceous structure of the fine mineral particles, which are, however, for the most part, of greater than colloid dimensions. The flake form of the particles would give them relatively enormous surface, and the penetration of water between the flakes is assumed to account for the tremendous swelling power of the clay when water is added to it.

Experimental Data

The following data was gathered in endeavoring to establish methods for simple determinations of the relative adsorptive

power of Montana bentonites as compared with that of a standard English fullers earth which, because of its high adsorptive power, has considerable commercial value.

Samples of material used in this work will be referred to by number for purposes of identification. They are: No. 1, English fullers earth; No. 2, Lincoln Mining Company bentonite; No. 3, Anaconda Highway, white bentonite; No. 4, Glasgow, Mont. bentonite; No. 5, Nissler weathered volcanic ash; No. 6, Anaconda Highway, brown bentonite.

Adsorption of Dyes from Solution by Bentonite

The standard solutions of all the dyes used consisted of about 0.125 grams of dye diluted to 1000 cc. with distilled water. Twenty-five cc.'s of each standard dye solution was shaken for 12 minutes with a 2.5 gram sample of bentonite and filtered. This procedure was repeated with 0.5 grams bentonite because in some cases practically all color was removed from the dye by the 2.5 gram samples of material. A blank was run with each standard dye to determine the amount of dye removed by the filter. The data obtained is summarized in the following table.

TABLE I

Amount of dye removed from solution by different bentonites.

Dye	cc.'s of standard dye sol. used.	Grams of bentonite used.	Order of re- moval of dye by bentonite	Effect of filter paper on dye
Methyl Violet	25	2.5	Almost complete by all samples	Large amount removed
	25	0.5	3,4,1,2,5,6	" "
Eocine Red	25	2.5	Slight removal by all samples	Very small amount removed
	25	0.5	" " "	" " "
Congo Red	25	2.5	1,2,3,4 almost complete; 5 & 6 about 75%	Small amount removed
	25	0.5	1 & 2 good; 3 & 4 fair; 5 & 6 poor.	Small amount removed
Aniline Orange	25	2.5	1,5,6,2,3,4	Small amount removed
	25	0.5	1 & 2 fair; 3 to 6 poor	Small amount removed
Malachite Green	25	2.5	almost complete by all samples	Small amount removed
	25	0.5	1,3,2,4,6,5	Small amount removed
Aniline Red	25	2.5	Almost complete by all samples.	Small amount removed
	25	0.5	1 excellent; 2 & 3 good; 4, 5, & 6 fair.	Small amount removed
Nigrosine	25	2.5	1, 2, 3, 4, 5, 6	No dye removed
	25	0.5	1, 2, 3, 4, 5, 6	No dye removed

Apparently nigrosine is the only one of the dyes used, that gives a fair idea as to the adsorptive power of the different materials because there was enough dye present in all samples after treatment, to show a gradual decrease in amount of dye removed between samples 1 and 6. The expression of adsorptive power by nigrosine seems to be in agreement with the expression by adsorption of acetic acid by bentonite, as is described below.

Adsorption of Acetic Acid from Solution

Before going into this part of the work, it may be well to discuss some of the principles involved in the phenomena called adsorption.

Solids possess a capacity of holding, on their surfaces, a layer of gas or liquid with which they come in contact. A loose chemical compound may be formed or the attraction may be considered physical in nature. In either case there is probably attraction between positive and negative charges. In many cases the layer of adsorbed molecules is only one molecule deep and further adsorption ceases when the surface of the crystal lattice is covered.

The amount of adsorption is greater at lower temperatures and it depends on the nature of the adsorbing medium and the material adsorbed. In the case of solutions it also depends on the solvent, and an equilibrium is set up between the material in solution and the material adsorbed on the surface of the adsorbent.

It seems quite improbable that this phenomenon is chemical in nature because argon and other inactive gases which do not (as far as we know) enter into chemical combination, are

readily adsorbed.

The qualitative facts mentioned above find quantitative expression in the form of an empirical adsorption isotherm of Freundlich⁵, $x = Ky^n$, where x is the weight of material adsorbed by a given amount of adsorbent and y is the concentration of solution at equilibrium, K and n are constants for conditions of the adsorption operation.

Although the Freundlich formula has been applied with more or less success to all types of adsorption, its value as a true representation of a basic adsorption law has been questioned. In this work, Freundlich's expression is used as an interpolation formula showing the comparative adsorptive powers of bentonites, because it is believed to be applicable for all practical purposes.

Procedure

Into each of six Erlenmeyer flasks, 2.5 grams of bentonite were placed, after which measured quantities of N/4 acetic acid was added beginning with 1.5 cc. in the first, and doubling the amount in each successive flask. The volumes in each case, except in the last, were then increased to exactly 48 cc.s with distilled water. The contents of the flasks were agitated at regular intervals for about 12 minutes, after which they were allowed to settle and then filtered. Proportional amounts of the filtrates were titrated with N/10 sodium hydroxide.

The values of the concentration at equilibrium (x) and the amount adsorbed (Y) are plotted on coordinate paper (Figures 1 and 2), and the values of the slope (n) of the straight line equation and the intercept (K) are noted.

The largest source of error, other than in manipulation,

is in the plotting of the curve (best straight line) since errors of small magnitude cause a large variation in the slope and intercept.

The equation holds nicely in certain cases, at other times it gives results far from actual. The difficulty appears to be that there are a number of types of adsorptive forces, so that any equation which assumes a single type of simple nature cannot always account for the results obtained.

However the equation does express the relative difference in adsorptive power of the different bentonites in contact with acetic acid solutions as shown in figure 1.

The curves plotted illustrate that bentonite samples No. 1 and 2 adsorb more acetic acid than samples 3 and 4.

In the past, too great an attempt has been made to explain the phenomenon of adsorption upon a purely physical or chemical basis. Although it is true that in many cases an adequate explanation seems to involve only physical forces, as surface tension, electrical potentials, etc., in others a chemical basis, at least in part, is necessary to explain the facts. Much unnecessary labor has been wasted, and real progress in science has been retarded by the endeavor of each group to explain all of the facts upon one basis. At the present time it is recognized that adsorption probably involves both physical and chemical concepts at all times, and in specific examples there may be an emphasis of one type but not to the exclusion of the other. This development is as it should be, because modern scientific thought is gradually bringing together the fundamentals of the two sciences.

Clarification of Oils

Several samples of crude brown packing house lard were treated with English fullers earth and with the same Montana bentonites that were used in the experiments on dye adsorption. The material No. 2 (see dye adsorption) gave the cleanest and most odorless product when the brown lard was percolated through the different adsorbents. It has been reported that some packing-houses use considerable bentonite for clarifying and bleaching purposes and that there is no waste product, because they use the oil filled bentonite in making soap.

Determination of the relative ability of Montana bentonites to decolorize mineral oils by the contact process was accomplished by adding 5 and 10 gram samples of the dried, ground material to 50 cc. of oil, and slowly bringing the temperature to 150°C. At this temperature the mixture was agitated for 30 minutes and then filtered. As in the case above, the No. 2 material seems to have been the most efficient in decolorizing the oil and removing the impurities.

A sample of oil which had been run for about 30 hours in an airplane was filtered through bentonite No. 2 and the product sent to a reputable commercial testing laboratory for analysis. The report received from the testing laboratory stated that the oil filled all requirements of specific gravity, flash point, fire test, cold test and viscosity; was an S. A. E. No. 70 lubricating oil derived from Pennsylvania paraffine base crude oil well refined and entirely suited to airplane lubrication.

Although adsorption is important in decolorizing oils, it is the writer's belief that probably no good method will be devised for evaluating bentonites for oil refining by the use

of dye adsorption as a criterion. This is evident, as crude bentonites which have little bleaching value may have a very high dye adsorption in comparison to excellent acid-treated bentonites; as tests showed that although 200 mesh acid-treated bentonite adsorbed about four times as much malachite green as 50-mesh material from the same lot, the action of the two materials in decolorizing oil was nearly the same.

Effect of Heating on Colloidal Properties

Davis and Vacher⁶ have shown that regulated heat treatment differentiates the types of bentonite by placing a small amount of minus 10-mesh material in a porcelain crucible and heating in an electric muffle. The temperature was raised by intervals of 50°C., and the samples were placed in test tubes of water and shaken. Below 350°C. all samples absorbed water. One sample lost its colloidal property (was not affected by water) at 410°C.; others at 510°, 575° and 670°.

Data Necessary to Plot Figure 1. & R.

2.5 grams English fullers earth.

Volume of solution = 48 cc.

	cc. solution used	cc. NaOH		Conversion factor	48 cc. equivalent
1.	40	0.45	x	1.2	0.54
2.	20	1.1	x	2.4	2.64
3.	20	5.6	x	2.4	13.44
4.	20	16.1	x	2.4	38.64
5.	10	18.5	x	4.8	88.80
6.	10	40.0	x	4.8	192.00
	Standard acid =	42.7	x	4.8	204.96

	<u>X + Y</u>	<u>X</u>	<u>Y</u>
	Initial concentration in cc. of 0.1N alk. for 48 cc. of solution	Conc. of sol. at equilibrium in cc. alk. for 48 cc. sol.	Amount adsorbed in cc. 0.1N alk.
1.	6.40	0.54	5.86
2.	12.81	2.64	10.17
3.	25.62	13.40	12.22
4.	51.24	38.60	12.64
5.	102.48	88.80	13.68
6.	204.96	192.00	12.96

	<u>Log X</u>	<u>Log Y</u>
1.	9.7324 - 10	0.7679
2.	0.4216	1.0073
3.	1.1271	1.0871
4.	1.5866	1.1017
5.	1.9445	1.1361
6.	2.2833	1.1126

Data continued:

2.5 grams bentonite--Lincoln Mine.

Volume of solution = 48 cc.

	cc. solution used	cc. NaOH required		Conversion factor	48 cc. equivalent
1.	40	2.2	x	1.2	2.64
2.	20	3.5	x	2.4	8.40
3.	20	8.2	x	2.4	19.68
4.	20	18.5	x	2.4	44.40
5.	10	19.9	x	4.8	95.52
6.	10	39.4	x	4.8	189.12
Standard acid =		42.7	x	4.8	204.12

	$X + Y$	X	Y
1.	6.40	2.64	3.76
2.	12.81	8.40	4.41
3.	25.62	19.68	5.94
4.	51.24	44.40	6.84
5.	102.48	95.52	6.96
6.	204.96	189.12	15.84

	Log X	Log Y
1.	0.4216	0.5752
2.	0.9242	0.6444
3.	1.2940	0.7738
4.	1.6474	0.8351
5.	1.9801	0.8426
6.	2.2767	1.1997

Data continued:

2.5 grams bentonite--Lincoln Mine (check sample)

Volume of solution = 48 cc.

	cc. solution used	cc. NaOH required		Conversion factor	48 cc. equivalent
1.	40	2.0	x	1.2	2.4
2.	20	3.5	x	2.4	8.40
3.	20	8.3	x	2.4	19.92
4.	20	18.5	x	2.4	44.40
5.	10	19.7	x	4.8	94.56
6.	10	40.0	x	4.8	192.00
Standard acid =		42.7	x	4.8	204.96

	$\frac{X + Y}{}$	$\frac{X}{}$	$\frac{Y}{}$
1.	6.40	2.40	4.00
2.	12.81	8.40	4.41
3.	25.62	19.92	5.70
4.	51.24	44.40	6.84
5.	102.48	94.56	7.92
6.	204.96	192.00	12.96

	$\frac{\text{Log } X}{}$	$\frac{\text{Log } Y}{}$
1.	0.3802	0.6021
2.	0.9243	0.6444
3.	1.2993	0.7559
4.	1.6474	0.8351
5.	1.9757	0.8987
6.	2.2833	1.1126

Data:

2.5 grams bentonite--Anaconda Highway.

Volume of solution = 48 cc.

	cc. solution used	cc. NaOH required		Conversion factor	48 cc. equivalent
1.	40	3.2	x	1.2	3.84
2.	20	4.0	x	2.4	9.60
3.	20	9.3	x	2.4	22.32
4.	20	19.6	x	2.4	47.04
5.	10	20.5	x	4.8	98.40
6.	10	41.4	x	4.8	198.72
Standard acid =		42.7	x	4.8	204.96

	$\frac{X + Y}{}$	$\frac{X}{}$	$\frac{Y}{}$
1.	6.40	3.84	2.56
2.	12.81	9.60	3.21
3.	25.62	22.32	3.30
4.	51.24	47.04	4.20
5.	102.48	98.40	4.08
6.	204.96	198.72	6.24

	$\frac{\text{Log } X}{}$	$\frac{\text{Log } Y}{}$
1.	0.5843	0.4082
2.	0.9823	0.5065
3.	1.3487	0.5185
4.	1.6725	0.6232
5.	1.9930	0.6107
6.	2.2982	0.7952

Data:

2.5 grams bentonite--Weathered volcanic ash from Nissler.

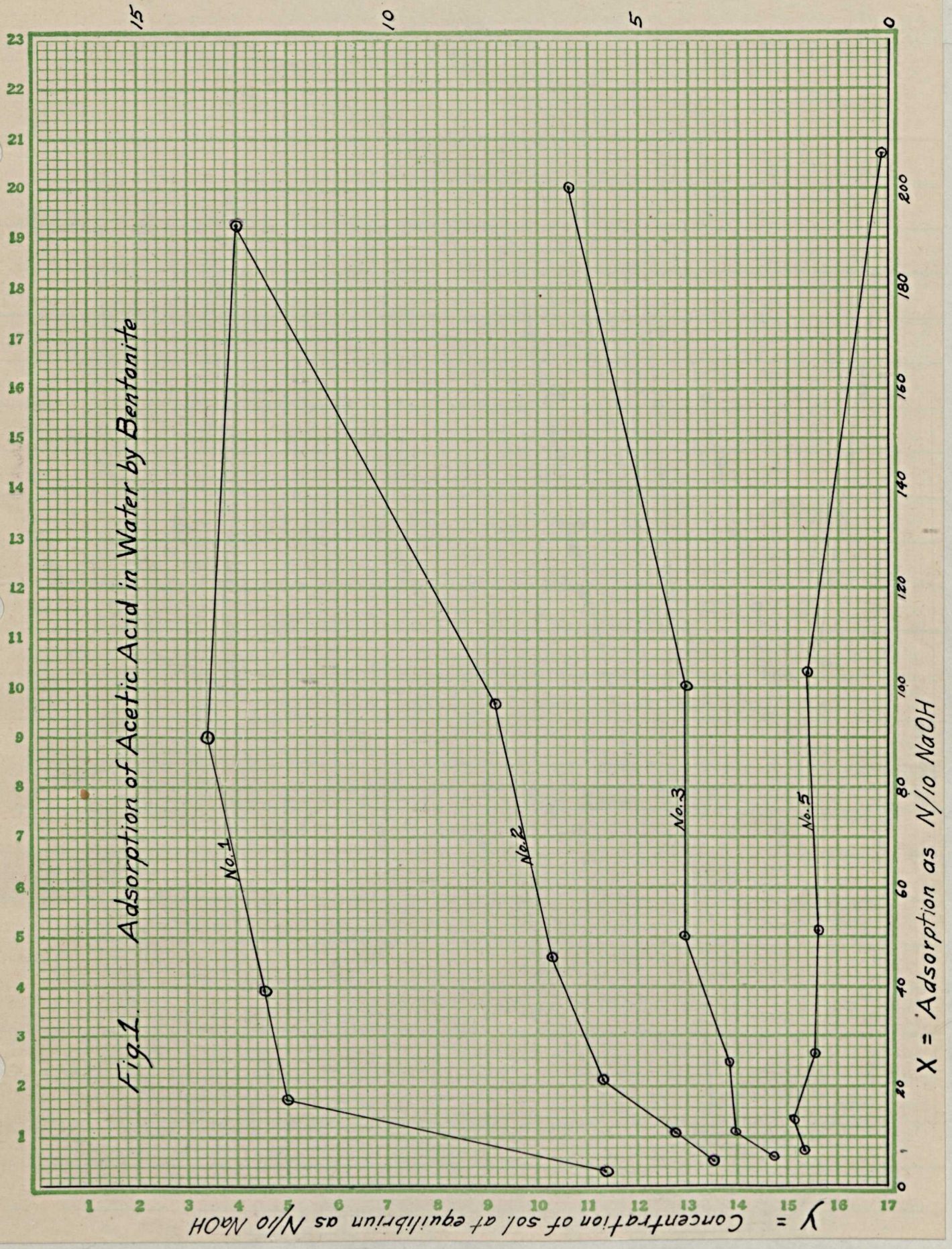
Volume of solution = 48 cc.

	cc. solution used	cc. NaOH required		Conversion factor	48 cc. equivalent
1.	40	3.7	x	1.2	4.44
2.	20	4.5	x	2.4	10.80
3.	20	10.0	x	2.4	24.00
4.	20	20.7	x	2.4	49.68
5.	10	21.0	x	4.8	100.80
6.	10	42.7	x	4.8	204.96
Standard acid =		42.7	x	4.8	204.96

	$\frac{X + Y}{}$	$\frac{X}{}$	$\frac{Y}{}$
1.	6.40	4.44	1.96
2.	12.81	10.80	2.01
3.	25.62	24.00	1.62
4.	51.24	49.68	1.56
5.	102.48	100.80	1.68
6.	204.96	204.96	0.00

	$\frac{\text{Log } X}{}$	$\frac{\text{Log } Y}{}$
1.	0.6474	0.2923
2.	1.0334	0.3032
3.	1.3802	0.2095
4.	1.6962	0.1931
5.	2.0034	0.2253
6.	2.3115	0.0

Fig. 1. Adsorption of Acetic Acid in Water by Bentonite



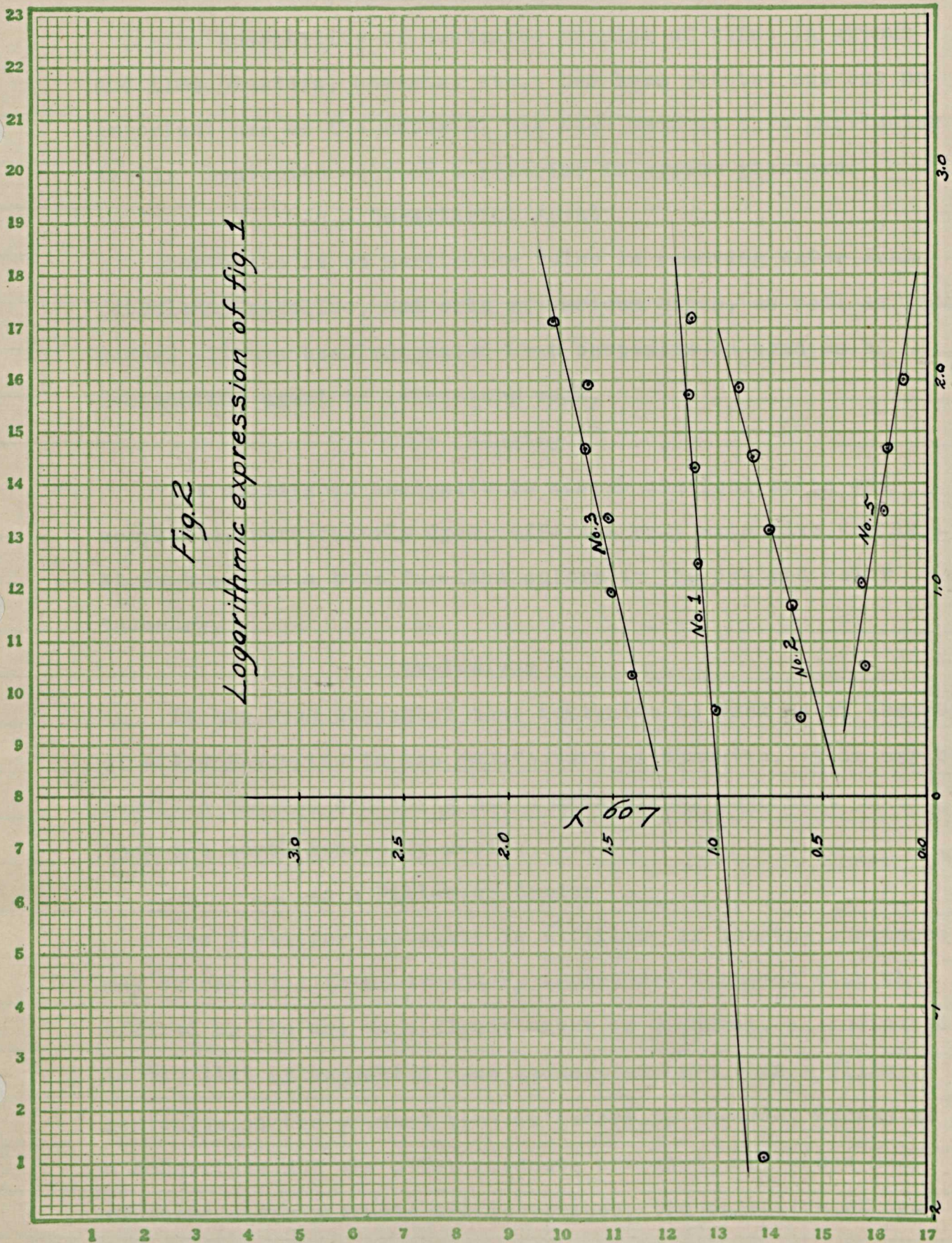


Fig. 2
 Logarithmic expression of fig. 1

1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17

Uses of Bentonite

Plasters and Cements

The possibility of bentonite proving of value in gypsum and lime plasters, cements, etc., has been pointed out but no important use of the material has been made in this particular field. Bentonite acts as a retarder in gypsum plasters.

As a De-watering Agent

Bentonite is of particular value in removing water from petroleum, gasoline, and oils of various kinds, as well as from gases. It has been suggested that bentonite be used as a desiccating agent in laboratory work.

Dye Industry

The ability of bentonite to adsorb chemical salts and compounds, particularly dyes, offers a very extensive use as a mordant and as a base for lake colors in the dye and color industry.

Explosives

It has been suggested that bentonite be used as an absorbent of nitro-glycerine in the manufacture of dynamite. This may be possible because bentonite has a much higher absorbent power than the earth generally used.

As a Filler or Loader

The field for bentonite as a filling or loading substance may be regarded as of secondary importance compared with that offered by its use as a direct agent or co-agent, as indicated in the list of suggested uses enumerated here. As a filler, bentonite at the present time is generally regarded merely as a substitute for the other products now used; and since many of these other products are low-priced, the objection of cost is one that is immediately raised and helps to lessen the

interest in bentonite. Few industries are prepared to undertake the refining of the materials used in their plants and until bentonite is offered to the trade, little active interest is likely to be expressed by it.

The new development of sprayed rubber suggests the possibility of securing rubber-bentonite emulsions, and the obtaining of thoroughly homogeneous rubber-bentonite bodies by means of spray mixing.

Pulp and Paper

One of the most important fields on which progress has been made in the use of bentonite is the paper industry, though even here the present application is small.

The Forest Products Laboratory of the United States Department of Agriculture has demonstrated that bentonite is very efficient in promoting the retention of china clay used as a loader in the manufacture of paper. In commercial tests, the addition of a small amount of bentonite resulted in a material increase in retention, and at the same time improved the quality of the paper.

As a straight paper filler or loader, bentonite is reported to have been used in large quantities by a Denver mill, most of the early production from Wyoming was used for this purpose.

Refining of Oils and Fats

The possibilities of clays of bentonite type have attracted much attention from the petroleum industry in the last two or three years, and important advances have been made in its use in the contact process of refining and clarifying petroleum products.

The manufacture as well as the use of acid-treated clays for oil refining is covered by a number of patents, and the contact process of refining by means of such clays is employed by

various companies on a royalty basis.

In addition to the possibilities offered by the petroleum industry, there naturally suggests itself a further field for acid-treated bentonite in the clarifying and bleaching of vegetable and animal oils and fats.

Water Softening

The property possessed by bentonite of effecting base exchange, that is, of selectively adsorbing ions from salt solutions, has been utilized in the manufacture of water softeners. Two of the best known water softeners on the American market that act on this principle have employed South Dakota bentonite as raw material. Briefly, the process consists in converting the clay into granules, heating to destroy its colloidal character, and then re-hydrating with a dilute solution of an alkali. On passing water containing calcium or magnesium salts through the product, ionic base exchange takes place, the calcium or magnesium being adsorbed and the alkali freed to combine with the acid radical of the original salts. The product may be regenerated by passing through it a brine solution, whereupon the reverse action takes place, the calcium or magnesium being released to form soluble chlorides, which are removed by washing.

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