Laramie, Wyoming
December 27, 1943

Senator J. C. O'Mahoney
U. S. Senate Building
Washington, D. C.

Dear Senator:

Dr. H. D. Thomas, Professor of Geology at the University and State Geologist of Wyoming, returning from a visit in Washington a few days ago, suggested to me that you may be interested in obtaining some additional information in reference to the fossil resin samples that he showed you while in Washington.

Guided by the desire of contributing a little to the general knowledge of this very important subject in our national economy, and especially of the West, I have taken the opportunity of preparing these enclosed notes.

As you may be aware, I am employed by the U. S. Bureau of Mines as Principal Consultant Metallurgist. I may assure you, however, that this work has been done in my spare time during the holiday season.

The expressed suggestions and criticisms are my personal opinions and do not represent or reflect the views of my superiors or of the U. S. Bureau of Mines.

The little time at my disposal in compiling this report may be the cause of errors and ommissions which I hope you will overlook. However, I shall be glad to be of further assistance to you in correction or clarification, and if you so wish I will develop this preliminary report into a more concise form, if requested through the U. S. Bureau or Mines.

Respectfully yours,

Dr. C. G. Flebus

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INTRODUCTION

USES AND PRESENT SOURCE OF SUPLY OF FOSSIL RESINS

FOSSIL RESINS

by Dr. C. G. Fiebus

Introduction.- Until recently the literature on fossil and natural resins has been limited. The varnish and plastics making industries have been hedged in by restriction and secretiveness. This report is an attempt to summarize the present knowledge on this subject with the scope of publicising the importance of some fossil resin deposits in the states of Wyoming and Utah, their role in the present war, and their possible contribution toward making America self-sufficient in another essential product.

USES AND PRESENT FOSSIL RESINS

Sources of Supply.- It is known that all the resin gums used in the United States, particularly those of the so called hardclass are now and always have been imported. Heavy tonnage has been used of superior quality in the making of high class varnishes, lacquers, shellacs, clear plastics, such as amber toilet articles, electrical and motor insulations, and transparent pipe and cigarette holders, telephone receivers, phonograph records, fountain pen cases, pocker chips, bullet-proof window glazing for planes, etc. The poorer resin gums are used in making opaque plastics such as radio dials and castings, auto parts, cheap colored plastic articles for electrical appliances, house hold furniture, linoleums, etc.

Recently, the poorer type has been used in fabricating airplane bodies and fuselages, and for automobile bodies and fenders. These two applications alone at present and in the future may require thousends of tons of resins.

It is difficult without obtaining data from the War Poduction Board to quote the quantities used at present, and also it is unwise and incorrect to produce pre-war figures that represent the beginning of a new industry.

Up to December 1941, the principal sources of production and collection were as follows:

I. Low acid number resins, including Damar and East India types

A. Damar resins - spirit and oil soluble

1. Batavia
2. Singapore

B. Batu

1. Batu
2. Black East India
3. East India-Singapore packing (Kosak)
4. East India Macassar packing (Mirde)

II. Resins of high acid number originating in East Indies, Copal type.

A. Manila Resins.

1. Melengket or soft resins - spirit soluble
2. Loba or half hard resins - sprit soluble
3. Fossil or hard resins - spirit soluble and oil soluble

III. African fossil or semi-fossil - oil soluble

A. Congo

IV. New Zealand fossil or semi-fossil resins - sprit and oil soluble.

A. Kauri (from living kauri trees and old roots.)

B. Bush Kauri (from site of old forest and lignite deposits composed of scrapping and powder.

V. Russian Copal - represented by two types:

A. The Verkhne-Agdzhekend types used for plastics are found in veins as monomineral in the sand and clay inclusions of young coal deposits of the far Eastern region.

B. The Artem types (milky-white and orange grains) are found in soft coal deposits of Shkoov-kungarin, also in the far Eastern region. There also are other copal deppsits; however, no data are available.

VI. Ukrainian Montan Wax (by-product from heated fossil resins). Recently crude montan wax was used as a substitute for the imprted Brazilian

Carnauba wax. In Ireland Monu Wax was obtained from resin constituents in peat deposits.

ORIGIN OF FOSSIL RESINS

CHEMICAL ANALYSIS AND SOME PHYSICAL

PROPERTIES OF FOSSIL RESINS

Origin of Fossil Resins.- Magnified sections of resin particles in the lignite and fussian bands of brown coal and in bituminous coal have suggested a Gymnosperm origin. Two kinds of resinous materials are identifiable under the microscope: one in the form of globules and rodlets of "dark brown color" found in the woody structure of the coal, the other consisting of particles which vary in size from that barely visible with the microscope to as large as a fist. The latter is evidently the result of resinous matter oozing out of wounds of the coniferous trees which eventually formed the coal deposits.

The fossil resins of brown coals may also be divided into two other groups, namely: (a) the bitumen resins, and (b) the retinites. The "bitumen resins" are distributed homogeneously in brown coal, can only be obtained by extraction, and contain large quantities of waxes.

The "retinites" occur in the coal in the form of small inclusions, can be separated mechanically, and are generally free from waxes. The retinites are identical with amber in composition and behavior. The bitumen resins differ from amber in many respects.

"Resinate" can be regarded as a pathological excretion that has been separated from the living plant wilst. The bitumen resins have remained within the cell walls and have been coalified together with the plant material.

Effects of Resins on Coal Deposits.- The resins have a favorable influence on the explosibility of coal because they readily form volatile decomposition products upon absorbing oxygen from air, also their presence is often the cause of spontaneous fire in freshly mined coal.

Coal deposits carrying the highest resin content.- Coals from the Tertiary and Cretaceous periods have the largest resin content. Paleozoic coal is never high in resinous matter. Also all ranks of lignite, sub-bituminous, bituminous, and semi-bituminous coal are found with high resin content.

The United States Bureau of Mines has a large number of photographs of various coals of Wyoming, Utah, Montana and North Dakota in which the various types of resins included in those coals are clearly shown.

The coals with the highest resin content known are assumed to be from the Sunnyside mine in Utah and from the Kemmerer coal field of Wyoming, and are said to contain from 25 to 30 per cent resinous matter.

(See enclosed map showing known deposits of fossil resins in western regions of the U. S.)

<u>Chemical Analysis and some Physical Properties of Fossil Resins</u>.- The resins belong to the bituminous fraction of younger and older coals and indicate their presence by a yellow to brown coloration. The compounds involved are high molecular esters which are related to the fats, waxes and terpenes. Even at ordinary temperatures they tend to take up oxygen and they exert a clear influence upon the coke and tar formation and on hydrogenation. Their average composition is 80% carbon, 8% to 9% hydrogen and 9% to 10% oxygen. In general, fossil resins do not contain nitrogen and sulfur, which only occasionally have been found in small amounts.

The resins of some coals show a bluish fluorescence when exposed to the ultra-violet light of the qartz glass analysis lamp so that their distribution in the coal is well recognizable. The matrix surrounding the resin inclusions often is fiber coal, fusite, whose formation in this case may be caused by the resin content. The structure of coal resins often shows a coagulation structure similar to that of amber, which indicates that those resins may be considered a solidified gel.

Some of the contrasts found between two resins submitted for laboratory examination, one from Newcastle (State of Washington), one from Sunnyside mine (Utah), and another from Kemmerer, Wyoming were as follows:

Physical Characteristics	Newcastle Resins	Sunnyside, and Kemmerer, Wyoming Resins
Color	Light yellow to greenish yellow	Light brown
Light transmission	Transparent to translucent	Transparent
Hardness	Scratched by crystal of Cu Sulphate	Powders easily
Specific gravity at 22°C	1.03	0.9887
Melting point	395°-402° C	98° - 101°C

These resins are insoluble in alcohol, but are soluble in varying degree (2% - 31.2%) in turpentine, ether, toluene, acetone, etc.

Another study on resins in coal from Western America, viz. Coalmont, B. C., reported that these resins would scratch Baltic amber, had a specific gravity of 1.03 to 1.05, and melting points of 210° to 390° C. Of two samples submitted, the first resin was soluble in alcohol, while the second was insoluble in alcohol but soluble in benzol. Both were soluble in turpentine. The resins were classified as retinites.

PRELIMINARY TREATMENT METHODS

FOR THE RECOVERY OF FOSSIL RESINS FROM COAL

Treatment Methods.- The average coals of Wyoming appear to contain approximately 5% resins in coal seams of 10 to 15 feet thick. The deposits are generally located in the southwestern part of the state in the vicinity of Carter, Kemmerer, Evanston, and south of Granger, but also near Sheridan.

It is difficult to estimate the tonnage of resinous matter, however, without fear of exaggeration. All the western coals may contain hundreds of millions of tons.

The specific gravity of the resins ranges between 1.01 and 1.075. The lowest specific gravity of coal ranges between 1.28 and 1.32. This permits the separation of the resins from coals by the sink and float method. In the sink and float method, the coal once wtted by a salt solution, containing one part in a million of tannic acid, will sink and the resins will float. The resins obtained can be separated and graded in several tyes by the same process but using a stronger solution, one part in 100,000.

The light resins, having a specific gravity of 1.01 to 1.05, are light in color, ranging from white to lemon-yellow. The resins with a specific gravity of 1.05 to 1.07 are dark in color, ranging from deep red to brown or black. The dark color in resins is due to the presence of asphalt. The asphalt can be removed from the resins by dissolving the resins in suitable solvents, such as naphta, or benzol, and adding a precipitant for the asphalt, such as alcohols, ether or acetone.

The resins separated by the sink and float method contain about one per cent wax. The waxes have a specific gravity below 1.00 nd will float on water. Resins, having a specific gravity higher than one, must be wetted by tannic acid, one part in 100,000

Successful separation of fossil resins from coal with a recovery up to 96% of the resin content, has been accomplished also by using sodium chloride or zinc chloride solution having a specific gravity of 1.10. The resins recovered were comparatively free from coal.

Froth flotation process using extremely small quantities of amyl alcohol has given high recovery of resins free from coal. This process was adopted in recovering commercial resin products from Salina Canyon Coal in 1936.

The combined Metal Reduction Co. (Brit.) removed resins from coal by froth flotation. Finely powdered coal is mixed with water to form a paste, a frothing agent is added(such as AmOH, turpentine, pine oil or creosol) and the mixture is subject to froth flotation.

The floated resins are separated and freed from the coal particles by reflootation in presence of K alum and additional AmOH.

It is clear from the above known methods of coal treatment for the recovery of its resin content, that neither expensive equipment or strategic reagents are required. Wood or concrete vats and wooden boxes are the only equipment required for the separation of resins from coal for a small scale operation.

THE POSSIBLE INDUSTRIAL USES OF FOSSIL RESINS IN THE VARNISH & PLASTIC INDUSTRIES.

INDUSTRIAL USES OF FOSSIL RESINS

Varnish Manufacture.- For the manufacture of varnish it is necessary to establish the solubility characteristics of the resins as well as their physical properties, such as color, light transmission, hardness, specific gravity, melting point, acid number, saponification number, iodine number and ultimate chemical analysis.

With these characteristics as a basis a proper process may be established for manufacturing varnishes for various indoor or outdoor uses.

Varnishes were made according to a prescribed receipe, by melting samples of fossil resins from Sunnyside, Utah, and adding hot boiled linseed oil and small quantities of litharge as a drier, and thinning the solution with spirits of turpentine. After filtering and alloing to settle for some time, the varnishes were tested and found to compare favorably ith some typical commercial varnishes.

They gave very satisfactory results, drying in the usual time with lustrous and hard surfaces. The only unfavorable feature as the rather dark color.

Samples of graded resins were submitted to various varnish manufacturers, though not in sufficient quantities for a plant test. They were unanimously recognized as good bases for making good varnishes. Many, in certain cases, could not compete with the large sized and much paler copals such as Zanzibar, Kauri, Manila and Congo Gums. However, certain resins from Lincoln and Uinta Counties, Wyoming, if properly graded, may be superior to the above enumerated imported resins.

The dark varnishes poduced from dark fossil resins cuid compare favorably in hardness with varnish roduced from kauri scrapping and dust, which is impossible to obtain today.

Cold Processed varnishes.- While investigating economical uses of the fossil resins in the paint and manufacturing industry, a cold process was tried. Varnishes were prepared by reflexing equal parts of toluol and resins. Fifty grams more of toluol was then added to the thin solutions and the undissolved residue then removed by centrifuging.

The percentage of resins in the solutions of three samples were A. 31%, B. 29.4% and C. 29.8%, showing an average of 10 percent of the resin insoluble in the toluol.

Next, to 60 parts of resin solution, 40 parts of heavy boiled linseed oil and one part of drier solution was added. No heat was used. The product was 20 gallons of cold processed varnish. Films of the varnish produced were spread on black iron panels by spinning, the same procedure was made with varnish produced from commercial Ester Gum. When dry, all panels were exposed on a roof of the laboratory at an angle of 45° to the vertical.

The condition of the films after nine weeks was recorded as follows:

Coal resins

A	Dull. Numerous checks, some lifting of films.
B	Dull. More checks than A, lifting of films.
C	Entire film rusted and peeled off.
Ester gum	Considerable loss of gloss. More checks and lifting than A. and B.

Varnish made by cold process similar to A and B might be utilized for dark coating materials.

In conclusion, the Western American fossil resins if properly graded and treated in accordance with the standard of the Mint Manufacturing industry, might be used as substitutes for all the varieties of resins and gums imported from Asia and Africa for the production of varnish, paints, enamels, and shellacs.

Amber Oil.- If distilled, fossil resins will produce an oil of similar properties to Amber Oil and which could be used as glue cement and in the production of plastics.

Moulding Process and Pastic.- Moulded articles could be made from coal resins by the use of pressure and temperature in combination.

Large sized blocks of amber were formed from fossil resins by heating the material to 200-250° C. and applying a pressure of 400 atmospheres. The dielectric properties of this material could be used to advantage and supplimenting products made with synthetic resins as Bakelite, Redmol, Condensite, etc.

Fossil resins could be modified by chemical action to resins of different physical characteristics for uses in the plastic industry for manufacturing transparent objects, like synthetic bullet-proof glazing for plane windows, transparent protective shields for police or military motorcycles, etc, or to produce thermoplastic materials.

In Germany, shortly before the war, a synthetic plastic named "Kolonite" was obtained by treating lignite with cresylic acid. This moulding compound posses the physical properties of Bakelite and allied substances. The material is machinable and takes a high polish. The application of such a process or a similar one to our own lignite, or even more advanced sub-bituminous coal, might be as well worth while.

Bituminous coal itself might be acted upon, let us say, in the plastic state by chemical reagents such as formaldehyde in gaseous phase to yield synthetic resins. At that time, the coal is in state of incipient decomposition. The phenolic substances are present and also the ammonia which is often used to catalyze the condensation of aldehydes with phenols as in the Bakelite process.

Conclusions.- While there is a large field of applications for fossil resins in the varnish manufacturing industry, the plastic industry is, however, the most sensible outlet for considerable quantities.

Large production is visualized if a treatment plant for recovery of fossil resins from Wyoming coal is built in conjunction with or as a subsidiary unit to a hydrogenation or low carbonization coke plant, because the powdered coal tailings present the best raw material for the two above said processes.

MARKET

Present Market.- The American Paint Journal of 1943 gives a price of $180 per ton for imported resins, Grade B, which have similar characteristics of Wyoming and Utah fossil resins.

The Hercules Powder Co., Wilmington, Delaware, before the present war, advertised the possibilities and properties of Stay-bellite resins (Hercules Hydrogenated resins) the price given per pound in lots greater than 500 lbs. was from 8 to 9 cents, less than 500 lbs, at 30 cents a lot.

Large quantities of synthetic resins are at present produced as by-products of the petroleum refining industry. They cannot supply the need of the country, however, either in ti e of eace or in time of war.

It is clear that in the afterwar period in a world of plastics (House trimmings, furniture, automobiles and planes) only the fossil resins obtained from coal could satisfy the market demand.

Steps should be taken now so as to developand protect this known, although new, source of income and industrial independence.

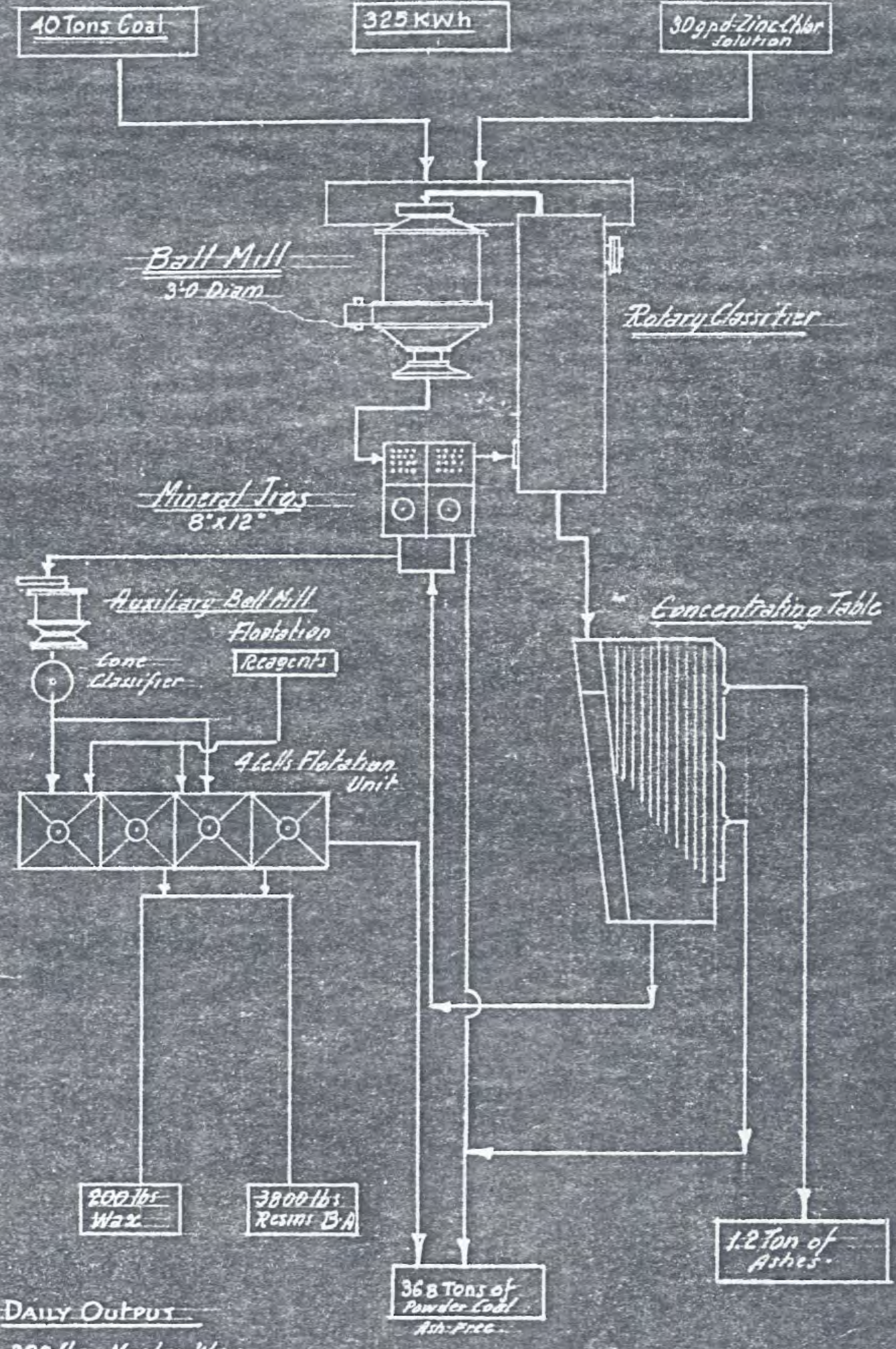
Present production cost in a pilot plant is $0.043 per pound. It is visualized that in a large plant the cost of recovery of fossil resins from coal may be $0.015 per pound.

PROPOSED PROJECT FOR AN

INITIAL DEVELOPMENT OF

THE FOSSIL RESINS OF WYOMING AND UTAH

PRELIMINARY-DAILY FLOW DIAGRAM for the RECOVERY of FOSSIL RESINS from BROWN COAL



DAILY OUTPUT

200 lbs - Montan Wax

3800 lbs - Resins Class B & A

36.8 Tons - Powder Coal - ash free

By Dr. C. G. Flebus

PROPOSED PROJECT FOR AN INITIAL DEVELOPMENT

To outline a tentative development, it is desirable that an examination and survey be made immediately to determine the folloing:

1. Coal fields of the Western region bearing fossil resins.

2. Determine the percentage of the resinous matter content specifying the physical properties of each specimen as follows:

a) Color
b) Light transmissions
c) Hardness
d) Specific gravity
e) Melting point
f) Acid number
g) Saponification number
h) Iodine number
i) Ultimate Chemical Analysis
j) Medium in which solute best
k) Solubility in known resin solvents.

3. Determine the approximate tonnage reserve for each deposit.

4. Since there are known deposits at Kemmerer, Uinta, Sheridan, New Castle, Wyoming, and Sunnyside Utah which contain fossil resins, a small treatment plant for roducing about two tons er day of finished product is recommended.

5. In Laramie, educational center of the state of Wyoming where present laboratory facilities may be useful, an experimental station should be established to determine industrial economical processes. The products obtained from the various treatment plants may be used here in making all known varieties of plastics and varnishes.

For the prosecution of the above outline program it is desirable that the U. S. Bureau of Mines be recommended.

Technical experience and data already in their hands should allow this governmental agency to carry out the project in the most economical manner.

A tentative preliminary estimated cost of this project is given in the the following chapters of this report.

PRELIMINARY ESTIMATED COST OF SURVEY

For the general survey of coal deposits bearing fossil resins

4 Mining Engineers at $3800 per annum for 4 months	$ 5070.
4 Mining Engineers traveling expenses " " "	3200.
General expenditures, stationary, etc,	1730.
Total exploration cost	$ 10,000.

For laboratory work, analysis and physical tests.

1 Chemist $ 3800 per annum 6 months	$ 1900.
1 Chemist Asst. $3200 per annum 6 months	1600.
1 Physicist $4200 per annum 6 months	2100.
1 Physicist Asst. $3200 per annum 6 months	1600
General Office expenses and laboratory materials	2800.
Total laboratory cost	$ 10,000.

General supervision and overhead expenditures.

1 Principal Mining Eng. $5600 per annum 6 months	$ 2800.
Traveling and overhead expenses for electrical work, pringing of informations reports and data, correspondence, etc.	2200.
Total supervision cost	$ 5000.
Total cost for a preliminary survey	$ 25, 000.

The sum of $25,000 is required to determine the potentiality and reserve of the U. S. fossil resins in the Western Region.

PILOT TREATMENT PLANTS

With the aim of obtaining some useful products in economical quantity and in the meantime that could support the expenses of the research, it is desirable that the proposed pilot testing plants be on a semi-commercial scale.

One part of the production could be sold to private concerns, the other could be shipped to the proposed Laramie Plastic and Varnish testing laboratory for experimentation.

The pilot plants should be designed so as to permit future additions and expansions, for the reasons already stated in this report, and should be complimentary units to some other industry where powder coal is the base as fuel or as raw material. Such a combination would result in a prosperous enterprise.

Proposed Typical Pilot Treatment Plant.- A few preliminary tests should be made using the present laboratory facilities of the U. S. Bureau of Mines in Salt Lake City before attempting to desing a pilot plant. However, for the purpose of establishing a preliminary estimate of the expenditures involved in the whole program a tentative plant design is here outlined.

The plant is designed to operate on a 24 hour per day basis with a minimum treatment capacity of 40 tons of coal per day. The estimated output of 2 tons of finished product per day is based on the assumption that the coal may contain 6% of resinous matter, of which about 80% is recoverable through the proposed process.

For economy, all operations are arranged in gravity flow circulation. The mixed coal stored in a wooden bin above a ball mill is fed into the mill by a chute, then the wet grinding is passed through a classifier and fed into a pulsating jig where some of the resins are separated from the coal. The tailings from the classifier are passed into a concentrating table. The resins recovered from the jig operations and from the table are sluiced into 4 flotation cell units where they are separated and graded in accordance with color and specific gravity, then stored in separate piles to dry and are made ready for shipment. The final product from storage piles is sacked in a separate building, weighed and shipped to the railroad by truck for final disposal to its destination. The tailing from flotation cells and concentrating table being mostly coal is then stored in a separate storage pile for future use. Wooden buildings are needed to house the equipment and to serve as an office and as a testing laboratory.

Preliminary Estimated Cost of Pilot Plant

Building Construction

Mill 20'-0" x 60'-0" x 20'-0"	at $0.15 cft.	$ 3600.
Shipping Office 15' x 40' x 14'	at $0.15 "	1260.
Unforeseen		1140.
	Building Cost	$ 6000.

Equipment

1 coal storage bin	$ 500.
1 Rotary weight feeder	350
1 Ball Mill 3' x 2'	1600
1 Rotary Classifier	800.
1 Pulsating Jig - two cells 8" x 12"	900
1 Concentrating table	600.
4 flotation cell units	1500.
1 10 HP Motor for ball mill	400.
1 2 HP " for the classifier	90.
1 ½ HP Motor for the jigs	50.
1 Wet Reagent feeder	245.
2 2 HP Motors for flotation cells	190.
2 1 HP for the concentrating table	90.
Placing and setting foundation and unforseen	2000.
	$ 9315

Office furniture and laboratory Equipment	$ 1185
Total equipment cost	$ 10,000.

<u>Total Construction Cost</u>

Building and Structures	6,000.
Equipment	10,000
Total	$ 16,000.

<u>Daily Operating Expenses</u>

<u>Labor</u>

1 Superintendent at $12.00 (one day shift)	$12.00
6 Laborers at $6.00 (2 for each shift)	36.00
1 Assayer at $8.00 (1 night shift)	8.00
8 workers	$56.00

<u>General Expenses</u>

40 tons of coal from local mines at $1.75 a ton	$70.00
Power 10 HP 350 KWH per day at $0.005	2.00
Flotation Reagent Pine oils, cresolic acid, etc.	5.00
Zinc chloride or sodium chloride for jig and mill	8.00
Depreciation of equipment	12.00
Office and general expenses	10.00
Total General	$107.00

<u>Total Daily operating expenses</u>

Labor	$ 56.00
General Expense	107.00
6% Interest of capital invested $16,000	2.68
5 years amortization of capital invested $16,000	8.90
Total daily expenditure	174.58

Assuming that the production is 2 tons of resins per day, the cost per pound is $ $\frac{174.58}{4000}$ = $ 0.043.

The present market value is $.09 per lb.; the estimated daily profit is $183.42. There will remain 38 tons of powdered coal that can be used for other purposes which may have a minimum value of $1.00 per ton, and makes an additional $38.00 to be added to the profit once a market is found.

EXPERIMENTAL PLASTIC LABORATORY

A plastic experimental laboratory is not only a useful institution for finding processes and economical methods of treating the fossil resins, but is a need long felt by the nation. Here small industries should be able to obtain data and information helpfull to them in surviving in a world of heavy competition made by big industries mushroomed by the war.

There are innumerable articles that could be produced in plastics by small commercial enterprises. However, they are impeded to do so because the processes or raw materials for producing them are controlled by big corporations with the aim of maintaining a closed and profitable market.

It is rather difficult in this preliminary outline to make an estimate of the cost of such an experimental laboratory. Equipment and plant layout require a careful study of all the various phases of processes now in use in making plastics and might require at least four or five months of intelligent planning.

However, for the sake of arriving at some approximate cost in starting such a project, I will say in view of past experience in the layout of other similar projects, I believe that $250,000. is sufficient to provide a well equipped laboratory and for a testing and producing plant.

PRELIMINARY ESTIMATED COST OF FOSSIL RESINS PROJECT

General survey	$ 25,000.
5 Pilot Plants in semi-commercial scale at $16,000 each	80,000.
1 Plastic Experimental laboratory	250,000.
Unforeseen	45,000.
Total cost of project	$ 400,000.

Comments

In view of the overwhelming demand for plastics in the afterwar period, the $400,000 spent today would return many fold this investment in new findings, and in the development of new industries in the west where our returning soldiers might find a permanent gainfull employment.

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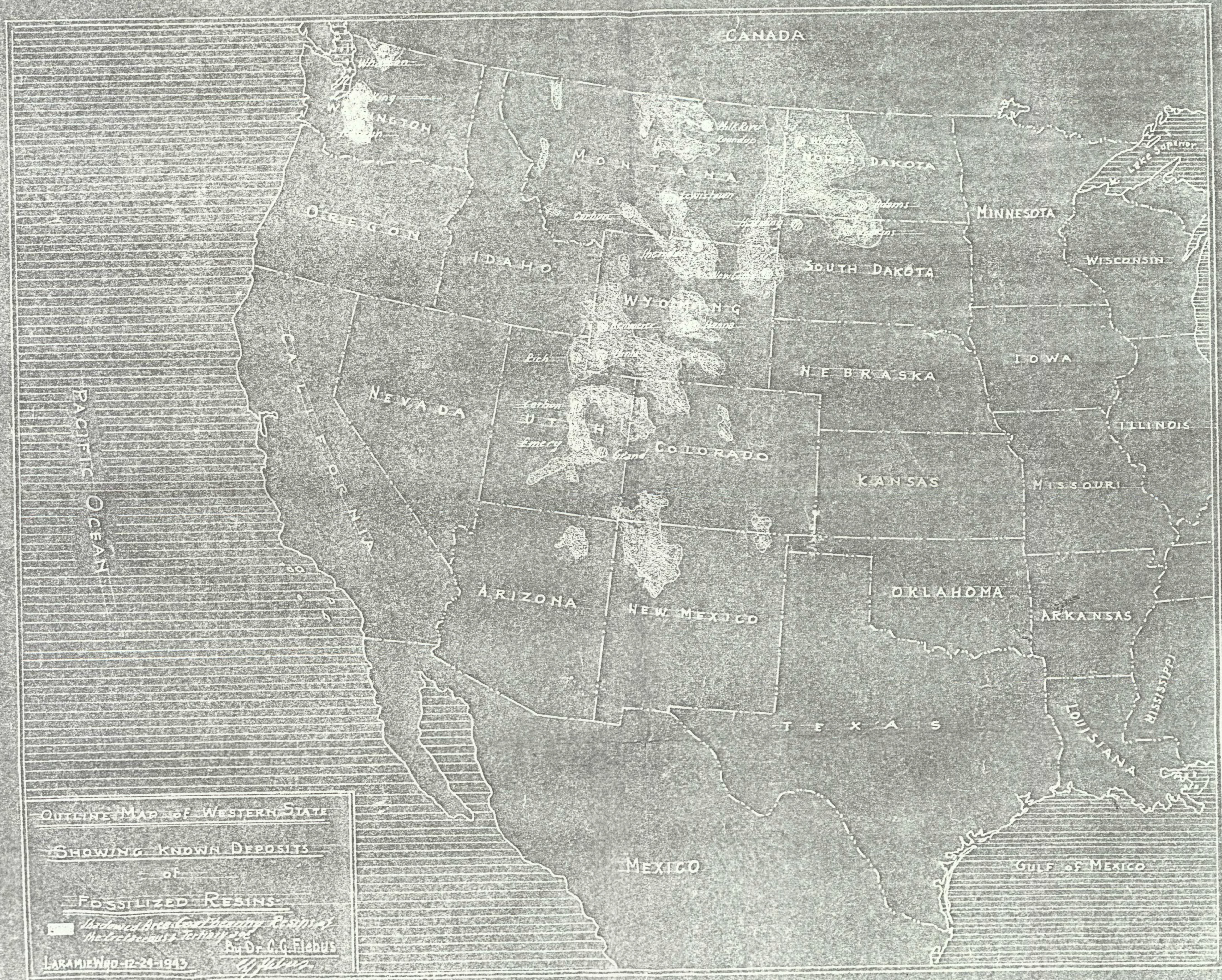
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UTAH
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NEBRASKA
KANSAS
OKLAHOMA
TEXAS
MINNESOTA
IOWA
MISSOURI
ARKANSAS
LOUISIANA
WISCONSIN
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