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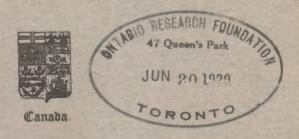
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THE HONORARY ADVISORY COUNCIL FOR SCIENTIFIC AND INDUSTRIAL RESEARCH

BULLETIN No. 7

THE MANUFACTURE OF ETHYL ALCOHOL FROM WOOD WASTE

G. H. TOMLINSON, B.A.



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OTTAWA, 1919

THE MANUFACTURE OF ETHYL ALCOHOL FROM WOOD WASTE.*

During the past ten years the conversion of waste wood into invert sugars for use in the manufacture of ethyl alcohol has achieved a measure of commercial success which unquestionably indicates that this will soon become a business of great and far-

reaching industrial importance.

Two such plants have been constructed in the United States both within the period to which I refer. These have been maintained in continuous operation throughout a considerable portion of this time producing the highest grade of ethyl alcohol at costs comparing favourably with those developed by any other process or source of supply. While it is true that no phenomenal results have so far been obtained, nevertheless an encouraging start has

been made and the trail seems blazed for further progress.

The process which has been used is based upon the now wellknown fact that when wood is heated under pressure with a dilute hydrolyzing acid a certain portion is converted into gluccse. Under such treatment it is now recognized that under a comparatively wide range of conditions as much as 25 per cent to 28 per cent of the anhydrous wood is rendered soluble. Of this amount as much as 80 per cent can be delivered in the form of fermentable sugar but to accomplish this requires most careful control. To exceed these results is apparently difficult and as far as my experience goes is not likely to be accomplished by the use of dilute acids. For the present, therefore, a conversion reaching a maximum yield of from 20 per cent to 22 per cent of fermentable sugars, or from 10 per cent to 11 per cent of ethyl alcohol corresponding to a maximum of 35 gallons of 95 per cent alcohol per ton of dry wood represents the immediate goal for which to strive. So far on large scale operations average yields have hardly exceeded 50 per cent of this amount, thus leaving a wide margin for further improvement before exhausting the possibilities which have already been opened up.

It is my purpose to briefly review the essential factors upon which commercial success has been based; also the lines along which my experience indicates improvements are likely to be made and then give a few figures bearing upon the actual results which

have been obtained.

About eighteen years ago Alexander Classen discovered that yields approximating the maximum, to which I have just referred,

^{*}Copy of paper presented at the first convention of Canadian chemists held at Ottawa, May, 1918.

could be obtained experimentally by heating comminuted wood under pressure using dilute sulphurous acid and at the same time maintaining a moisture ratio even below the saturation limit. This matter of low moisture ratio represents the essential point upon which the success of this business has so far hinged. illustrate this, let us consider what would result, assuming that an acid concentration of 1 per cent is to be used and in one case that we have a moisture ratio of 100 per cent and in the other a moisture ratio of 400 per cent. Under these conditions the total acid consumption in the second case would be four times. and the amount of heat used something in excess of three times that in the former. Since the acid must subsequently be neutralized the lime required for this purpose will bear a similarly increased When the reaction is completed, although the yields may for the sake of argument be identical, the sugars formed will be correspondingly diluted and probably require in the second case some concentration before being used. These points all represent direct economy without which the business could not have progressed, but apart from this there are other factors involved of equal, if not greater importance.

The reaction itself is one requiring extremely careful control, especially in connection with temperature and time conditions. This being the case the less water present the more quickly can temperature regulation be effected and upon such rapid control largely depends the yield which may result.

To all of this may be added the fact that with the 100 per cent moisture ratio the resulting product is still below the saturation limit and represents a material with which it is not at all difficult to deal. In this regard it can be handled in conveyors like the original sawdust and the sugars can be extracted by diffusion just the same as in the beet sugar process. On the other hand with a high moisture ratio the solids quickly settle down into a felt-like mass which nothing short of an hydraulic jet is likely to move.

Such success as has accrued is directly attributable to an appreciation of the full importance of the above factors, without which commercial results could not have been obtained. In addition to this the application of rational engineering methods to accomplish the various operations involved has been all that has been required. Classen's failure to establish his process commercially in the United States was principally due to the latter.

In 1909 while conducting the operations of an experimental plant, the construction of which I initiated, for the purpose of demonstrating that my deductions in regard to the above were sound as well as that the engineering adaptations which I proposed were feasible, I discovered that hydrochloric acid and sulphuric acid could apparently both be employed with similarly low moisture ratios and similar success.

It was fully appreciated at the time that the advantages were largely in favour of using a volatile acid since, by this means, a uniform mixture and penetration of the wood was assured, while, otherwise, mechanical admixture was necessary.

Nevertheless the difficulties in this connection, which the use of sulphuric acid involved, did not appear insurmountable and to avoid dangers of patent infringement, which otherwise would be faced, its use was from that time on employed and the development which has since occurred has proceeded accordingly.

In comparing the relative advantage of the three reagents it would seem that under certain conditions about the same yields can be obtained with each. The maximum yield which I have noted as a result of the vast number of experiments which have been conducted has approximated 23 per cent fermentable sugar. As a matter of fact the best yield of which I have a record shows 23.1 per cent fermentable obtained by using hydrochloric acid. Next to this is one showing 22.96 per cent obtained by using sulphurous acid. In this particular experiment two tons of sawdust were employed. In the case of sulphuric acid many small scale experiments have given as much as 22 per cent fermentable sugar, but large scale experiments have never, to my knowledge, exceeded 75 per cent of this figure although there have been a great number of such experiments made. I have, therefore, reluctantly been led to believe that using sulphuric acid a sufficiently intimate mixture and penetration of the wood by the acid cannot be obtained in any practical way to give the best results.

Since hydrochloric acid apparently offers no particular advantage and at the same time involves greater expense further development of the sulphurous acid process is naturally to be anticipated if my deductions in this connection are correct. By applying the same engineering principles, which the Ewen-Tomlinson, or sulphuric acid process, has already proved to be sound and of commercial application, success along these lines is unquestionably assured as was sufficiently demonstrated in 1909 in the experimental plant to which I have just referred.

The apparatus and equipment which has been thus employed has given thoroughly efficient and satisfactory results. In many details of course improvements may be made but the main principles embodied in the original adaptations I believe may be expected

to endure.

The digesters in which the conversion is made are standard 14-foot globe rotary bleaching boilers. These are protected on the inside with an acid proof tile lining similar to that customarily used in sulphite pulp digesters. This type and shape greatly facilitate filling and emptying, and also provide simple means for needed agitation. The acid, as well as the steam, are admitted through the trunnions. A turnover of from eighty to one

hundred tons of sawdust per day of 24 hours can be realized from each such unit, and one man with a helper on each shift can handle

the operation of several units.

The separation of the sugar from the woody residue is effected in a standard beet sugar diffusion battery provided with a lining similar to the digester linings. Here again great capacity results with minimum labour expense in addition to which any losses in extraction can be practically eliminated.

A great deal of course depends upon the provision of smoothly operating conveyor equipment, properly designed hoppers, chutes, etc., all of which are necessary for the economic handling of the large volumes of material involved.

The fermentation of the saccharine, distillation of the beer and rectification of the alcohol involve no new problems and standard methods and equipment are used throughout. To insure successful fermentation, however, not only must the hydrolysis be properly regulated to give the largest yield of fermentable sugar with a minimum of decomposition products, but even then undesirable secondary reactions are liable to occur during the subsequent steps.

The hydrolyzed product irrespective of the acid reagent used always contains a certain percentage of pyroligneous and intermediate products. These constitute what seems a complex, unstable and ill-defined mixture and it is here that the trouble The undue formation of these can, of course, to a considerable extent be controlled in the cooking operations and in small scale experiments difficulty through secondary reactions is not so likely to occur. In this case where the mass is small cooling is naturally rapid and conditions for continued chemical activity negligible. In dealing with large masses, on the other hand, unless special provision for cooling is made some, or all, may retain a temperature approximating the boiling point for considerable intervals of time and under these conditions further reactions proceed over which there is no control. This difficulty it would seem has been the cause of most irregular and varied output and so far it has apparently prevented the duplication on the large scale of yields approaching those thoroughly established in the laboratory.

I have more recently discovered, however, that by introducing the new and very simple step of applying a vacuum to the digester as soon as the primary reactions are completed this difficulty can be overcome. By this means the mass is quickly cooled and the further advantage is obtained of removing a considerable portion of the volatile organic acids, which are always formed, as well as any trace of sulphurous acid which might otherwise remain. This step is now a matter of patent record, and, I believe, will be found to represent one of the most important contributions to this art which has been made. The residue, after the sugars are

removed, amounts to about 70 per cent of the original wood. This has been satisfactorily used as fuel to supply the necessary steam and power for the various operations and in doing this serves a most important requisite for success.

Let us now consider for a moment the commercial possibilities developed. Unfortunately I am not in a position to present any cost figures with the exception of those developed during the first six months following the commencement of operations in each of the two plants which have been constructed. Following this they have not been under my control or management and any more recent figures which I have seen I feel that I must treat as confidential. A brief review however of this initial period may be of interest.

Construction of the plant at Georgetown, S.C., was commenced in September, 1909, and its operation began July 12, 1910. This plant was designed to give a daily capacity of two thousand gallons of 95 per cent alcohol based upon a minimum yield of 20 gallons per dry ton and represented a capital outlay of \$171,479. The average daily capacity realized to December 23 amounted to 675 gallons showing an average yield of 16.8 gallons per dry ton for the entire run. Manufacturing costs ranged between 29 cents and 19.63 cents per gallon. The latter figure was realized on an output of approximately 1,000 gallons per day, which was the maximum average obtained for any continued period. These figures cover material, labour, and all factory expenses and repairs but do not include interest, depreciation, insurance or management. These items were not properly accounted for but I estimate amounted to an average of 14 cents per gallon for the period in question.

Considering the rapidity with which the plant was thrown together and the parsimonious conditions which were imposed upon its design these results were principally disappointing in regard to the yield and this it was anticipated would rapidly improve. At the beginning of the year 1911 the plant was taken over by the DuPont de Nemours Powder Company and since then I have had nothing directly to do with its operation.

At the fifty-third meeting of the American Chemical Society held in New York, September, 1916, a telegram was read, which contained the following: "Trust that those interested in ethyl alcohol from wood waste realize that the process is a great commercial success and that the interest in this subject will grow accordingly." This message was reported to have been sent by Mr. J. Stuart Groves, who was represented as in charge of the Georgetown plant, and as far as I know, is the only public statement regarding the proposition which has been made by a member of the DuPont organization.

In December, 1911, a second plant was begun at Fullerton, La. This was designed along much more extensive lines with the objective of 5,000 gallons of 95 per cent alcohol per day. The capital expenditure to July, 1913, including a commission of \$52,658.15, which in no sense was a necessary item of the cost, amounted to \$509.578.71.

Spirits were first distilled January 22, 1913, and distillation was continued more or less regularly and at about one-quarter capacity until the middle of the July following. Yields, output and costs covering each month during this period are set out in the following table:—

Month.	Yield in Gallons per dry ton of sawdust.	Output Gallons.	Cost per Gallon.
			figures
January and February	12.6	18,026.7	not available
March		15,549.0	48.9
April	14.95	10,249.5	45.99
May	16.9	36,288.2	38.74
June		34,358.5	32.94
July		37.817.8	29.29

The average cost covering the production of the 152,189.7 gallons which was made, amounted to 36.48 cents.

Owing to the fact that a departmental system of costing was used an accurate summary of the makeup of these figures is difficult. Considering the month of June, however, as representative the following analysis, which I have made, is I believe approximately correct:—

Raw Materials and Labour.		1.
Wood waste at 50 cents per cord. Acid (H ₂ SO ₄). Lime. Malt sprouts. Molasses. Glue. Labour.	· 051 · 019 · 008 · 019 · 012 · 002 · 061	170
Other Items— Superintendence Supplies Repairs Power and steam Depreciation Insurance Factory expenses General burden	·014 ·013 ·019 ·040 ·032 ·004 ·008 ·003	•172
Difference between cost of products manufactured in May and used in June and actual June cost		·133
Cost per gallon.		•329

The item of power and steam covers the complete departmental power house expense including labour, superintendence, depreciation, etc., which accounts for its being so large. Malt sprouts and molasses were used in the preparation and manufacture of yeast.

The figures given above must, of course, be considered in relation to the very low average capacity and poor yield which was obtained in order to properly appreciate their significance, owing to the very large proportion represented by fixed charges, as well as labour, which for the same reason was inefficiently employed. Thus it was estimated by qualified experts that if a monthly capacity of 100,000 gallons was reached and maintained, together with the yield of 20 gallons per dry ton, that a similar cost not exceeding 21 cents per gallon would result. Since the maximum yield which was recorded in 1913 amounted to 22.6 gallons per dry ton and the maximum quantity of sawdust processed in any one day was 330 cords, equivalent to 207.9 dry tons, the attainment of such a result seemed by no means beyond the realm of reasonable expectation. Furthermore it was convincingly represented that the contemporary and equivalent cost of producing alcohol from grain in the largest American distilleries, running at full capacity. exceeded 30 cents per gallon.

The very low capacity which was obtained during these early months was largely owing to the fact that no market for the output had been developed and it was only towards the end of June that this condition was adjusted and a real increase in capacity could be considered. In face, however, of the noticeable and seemingly reasonable improvement which results were indicating operations were unfortunately completely suspended July 18, 1913. This step was taken owing to the fact that working capital which had been largely dissipated was seemingly insufficient to carry the project further. Since then, but under different management, the plant was operated for a few months in 1914 and since the summer of 1916 has been running, as I understand, continuously taking advantage of the high price for alcohol which has since prevailed.

Regarding the quality of the alcohol which was made at Fullerton the following report covering a sample taken from bulk is representative of several independent examinations which were made. "The sample was found to contain 94.7 per cent of ethyl alcohol by volume. It is practically neutral, contains no acetone, no formic or other esters, no furfural, only traces of aldehyde (which is always present in alcohol and soon forms again after being removed) and so little of higher alcohol (fusel oil) that their quantity could not be determined colorimetrically. In all I consider this as one of the purest Cologne spirits that have come under my observation."

Under to-day's conditions the capital investment involved in the construction of such a plant would of course be much greater than it was in 1913. On the other hand I have endeavoured to show that a much larger yield can be obtained in any new plant if full advantage of the experience and knowledge so far gained is properly applied. Thus if a yield of 30 gallons or more per dry ton is obtained which involves nothing beyond the duplication on the large scale of experimental results which have been thoroughly established, any such increase in investment, as well as increases in labour rates, and raw materials, which have since occurred, should be more than balanced. On the other hand the cost of producing alcohol from either grain or molasses has at least doubled since 1913, and with alcohol in its present demand it would seem that a more favourable opportunity in which to proceed with some further development along the lines which I have discussed is not likely to occur again.

In addition to the possibility of realizing unusual profit the saving of food materials which can thus be made is an item not to be ignored and should ensure the proposition receiving the

attention which it apparently deserves.

While the sugars thus made have so far been exclusively used in the manufacture of ethyl alcohol there seems no question but that, like cane molasses, they can also be used for feeding purposes. I am strongly of the opinion that there are great possibilities in this direction. So far this is something, however, which has apparently not been given the investigation and study which it demands and beyond the fact that a product can thus be made, which is acceptable to animals, there is little I can say from my own experience regarding its practical application.

There is no question, however, regarding the food value of the carbohydrates which can thus be obtained, nor regarding the low cost at which they can be produced. A cost of from 4 to 6 cents per gallon of such wood molasses I figure should be easily realized and this should be fully equivalent to a gallon of cane molasses for feeding purposes. As a means, therefore, of quickly adding to our food resources this is something which should not be overlooked and offers, as I believe, unusual and practically unlimited

possibilities for investigation and development.

In the brief time at my disposal it has been impossible to more than touch upon the essential factors in the development of this business with the early stages of which I was so intimately associated. I trust, however, that as a result I have been able to stimulate some wider interest in this particular field which so far has been only scratched and which offers such wide opportunities for further progress.

N.B.—Gallons refer to U.S. wine gallons and when referring to alcohol 95 per cent alcohol is always inferred if not specified.

APPENDIX.

Since writing the above, I have no information regarding any further developments of this process in either the United States or Canada.

It has been reported, however, that several plants for producing ethyl alcohol from wood waste, were installed in Germany during the war, and that their output of alcohol was relatively large. So far as I am aware, however, no details regarding these developments have been made public up to the present time.

Since hostilities ceased the alcohol market throughout the world has been in a most unsettled condition, and until peace activities are more completely resumed, further expansion can

hardly be expected.

The demand for industrial alcohol is, however, increasing steadily, and it is a safe prediction that it must soon be utilized to supplement our gasolene supply. For this reason it is worth considering to what extent this might be accomplished in Canada, by making use of our supplies of wood waste along the general lines which I have discussed.

It is claimed that in British Columbia alone, that the saw-mills produce from ten to fifteen thousand tons of wood waste every day. Conservatively estimated this would represent sufficient raw material to produce from thirty to fifty million gallons of alcohol per year, or enough to operate one hundred thousand motor cars.

With such a result entirely possible, it is therefore to be hoped, that some serious effort may soon be made to demonstrate and develop in Canada the extensive possibilities which this apparently

presents.

In this connection the first step might be for our Forestry Department to give the whole subject thorough investigation, and even go so far as to initiate a commercial demonstration if this should then be found advisable or necessary.

Before anything is likely to be accomplished, however, our antiquated regulations governing the manufacture and sale of alcohol must first be revised, so that alcohol can be made and sold for industrial purposes without the needless restrictions now

mposed.

Proper legislation of this kind would also stimulate industrial activity along many other chemical lines, which are now closed to development in Canada as a result of our short-sighted policy in this connection, and for which there is apparently no excuse now that potable alcohol ceases to be a major source of revenue in this country.

July 16, 1919.