

# BONE CHAR IN SUGAR REFINING

## ITS USE AND TESTING

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### INTRODUCTION

It will probably be the most convenient introduction to this paper if I describe in broad outline the manner in which bone char is used in a sugar refinery, before going on to a more detailed account of how it is made and tested.

Bone char is used for removing colour and ash from refinery brown liquor to give fine liquor, which is the primary feed to the fine sugar vacuum pans. It is used in cast-iron cylinders of 8–10 feet in diameter and 20–25 feet high, which contain 25–35 tons of bone char. These cylinders are called cisterns or filters and they have a perforated false bottom over which a sheet of canvas is laid which prevents the char from falling through whilst the liquor flows without hindrance.

When the de-colourizing and de-ashing capacity of the bone char has been largely used up, the sugar is washed out with water, the process being known as "sweetening-off", and then the washing is continued for a time to ensure that the absorbed ash is removed, this process being known as "washing-off". The char is allowed to drain in the cistern and is then transferred by conveyor belt and bucket elevator to the dryer section of the char kiln where the moisture content is reduced from 20–25 per cent to 5–10 per cent, and it then proceeds to the kiln pipes where the remaining moisture is removed and the char is raised to a temperature of about 400–450°C in the absence of air. The flow then continues through the cooler pipe, where the temperature is reduced to below 100°C still in the absence of air, and the regenerated char then passes from the cooler pipe through the draw mechanism and is ready to begin another de-colourizing and de-ashing cycle.

### HISTORY AND MANUFACTURE OF BONE CHAR

Bone char was first introduced in granular form in 1828, but due to the slow development of methods of regeneration, it was not until about 1890 that sugar refiners began to make effective use of this valuable aid to the refining process.

The method of manufacture of bone char is to de-grease cattle or horse bones and then heat them in the absence of air to 800–900°C. This process carbonizes the organic matter in the bones, yielding a product containing roughly 80 per cent calcium phosphate, 7 per cent calcium carbonate and 10–14 per cent carbon, of which about half is free carbon, and the remainder is contained in organic compounds.

Originally, the bones were carbonized nearly whole and crushed to size afterwards, but it was a very wasteful process due to dust formation, and nowadays the crushing and grading is generally carried out on the unburned bones and subsequent sieving to specification is done by rotating screeners.

Also at first the carbonization process was a batch-wise one and after heating for a specified time, the char was withdrawn into sealed canisters and allowed to cool in the absence of air, but nowadays the process has developed into a more or less continuous one.

Due to the delays already mentioned in the development of regenerating kilns, it was originally part of the business of the char manufacturer to regenerate char and thus it was only loaned to the refinery and was taken back for regeneration and re-loan.

In the early days, also, the char was generally used in much larger sizes than now and was sieved to a 2–4 meshes-to-the-inch specification. Even as recently as the First World War, an 8–12 mesh analysis was quite common, and in America, in one refinery, even in 1953, the specification was 6–20. Eventually refiners began to realise that by utilizing higher pressures to force the brown liquor through the char, a much finer grist could be used and this would greatly increase the surface area and enable much more effective use to be made of the char. The usual specification nowadays is 9–35 mesh, which generally allows a maximum of a few per cent larger than 9, or smaller than 35 meshes-to-the-inch and is rather more strict on the percentage finer than 60 mesh, since any appreciable amount of such fine char would materially affect the flow rate through the cistern.

### REGENERATING KILNS

#### (1) The Scotch Kiln

The char kiln is the most important part of the char cycle and in its essentials, the Scotch Kiln which is in use at Rossburgh, and which is illustrated in the diagram, comprises firstly a dryer section where the wet char, after washing, falls slowly over horizontal pipes, known as "Cathedral Pipes" because of their shape, through which hot flue gases are passing, and the surface moisture is so effectively removed that visual inspection would declare the char to be completely dry. At this point the material actually contains 5–10 per cent moisture and since

this is absorbed moisture, quite an input of energy is required to satisfy the heat of adsorption which must be supplied before the char becomes actually dry.

The char falls from the bottom of the dryer section and forms a cone at its own natural angle of repose which covers the tops of the kiln pipes to a distance of a foot or so. This acts as an air-tight seal, since the first occurrence when the char enters the kiln pipes is that the adsorbed moisture is driven off and this upward flow of steam effectively prevents any downward ingress of air.

The kiln pipes are 8—9 feet long and a study of the temperature gradient of the char as it traverses this tube is quite interesting.

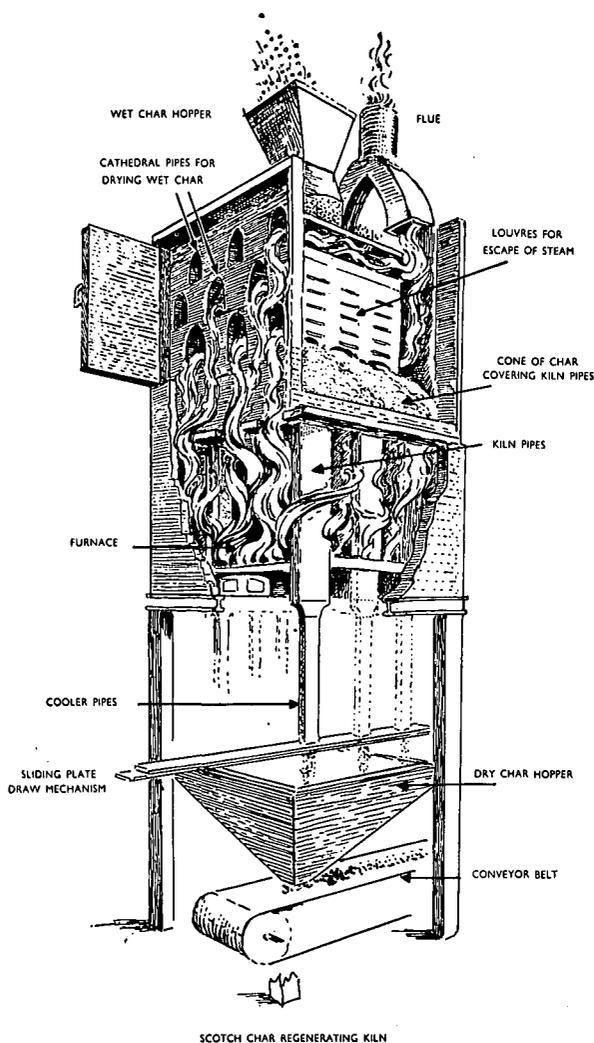


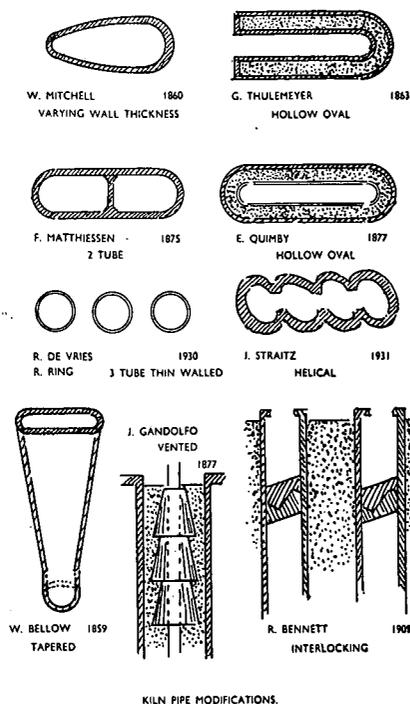
Fig. I

Initially the temperature remains at about 100°C while the water is being removed and roughly it is found that for the first 2-feet at the sides, and the first 3-4 feet at the centre, the temperature has still only risen to a little above 100°C.

After this, the temperature rises slowly at the centre of the pipe, and more rapidly at the sides, until the final maximum temperature of 400-450°C is usually reached at about 1-foot above the bottom of the kiln pipe and is maintained for about 1-hour, that is for about 2-foot depth in the pipes. This means that the maximum temperature region extends into the cooler pipe and thus it is of the utmost importance that the junction between kiln pipe and cooler pipe is air-tight.

The maximum temperature of the char in the centre of the pipe may be as much as 30°C below the maximum of the char at the sides because of the low thermal conductivity of char, and to minimize this disadvantage, a lot of experimental work has been done on kiln pipes of different shaped sections.

Some of these sections are egg-shaped with varying wall thicknesses, horse-shoe shaped, hollow ovals with the char in the annular space, oval with cross partitions giving a double tube, sets of three circular thin-walled tubes, in place of one oval-shaped tube, tapered tubes and some have internal vented louvres or cork-screw shaped strips running down the centre of the tube in order to prevent the char which starts at the side of the tube from remaining in that position throughout the length of the kiln pipe. These are illustrated in the accompanying diagram.



KILN PIPE MODIFICATIONS.

Fig. II

For a long time, the material of which the kiln pipes are made has been cast-iron, although cast

chromium iron was tried in 1922, its increased price being off-set because of the longer life and the higher temperatures that can be maintained. When it is realised that the conventional kiln pipe which has been in use at Rossburgh is between  $\frac{1}{2}$  and  $\frac{5}{8}$  inch thick cast-iron, some idea of the amount of heat absorption will be realised.

A start has been made recently to replace these completely by stainless steel tubing of 16-gauge thickness and of the same cross section as the internal cross section of the previously used cast-iron pipes. Stainless steel pipes give considerably less trouble since there is much less tendency to elongate and buckle with use and the maintenance fitter is very much better pleased at having to transport, erect and fit stainless steel kiln pipes weighing 15–20 lb., rather than cast-iron ones weighing 400lb.

One great fault with the cast-iron pipes is that cracks can occur which are not readily visible to inspection when the pipes are cold and yet are capable of admitting air when hot, thus causing decarbonization of the char. Another disadvantage is that at the temperature of the hottest part, carbon from the char can actually be adsorbed by the inside portions of the cast-iron pipe, and this is another method by which the carbon content of the stock Char can be fairly quickly reduced from its initial 12–14 per cent down to 5–6 per cent, and sometimes even lower.

Since no air is deliberately admitted during this regeneration process, it is obvious that the colouring matter adsorbed by the char can only be removed by a distillation process, and as anyone who has ever performed a distillation of a mixture of high molecular weight organic compounds will readily agree, some carbonization is almost bound to occur. It is for this reason that in many refineries, the carbon content of stock char is found gradually to increase and eventually it blocks the pores and makes the char less effective.

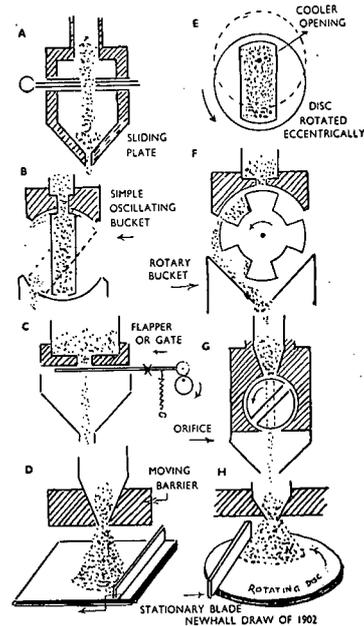
Even though it is carbon which is doing the colour removing, and the carbon content has been increased, this additional carbon is in an inactivated form and thus contributes little to the colour-removing property. There have been many designs for decarbonizers through which the char can be passed to reduce the carbon content to its optimum level, but the easiest method with most types of kiln is to allow a small controlled quantity of air to enter the cooler pipes and thus the decarbonization is effected at the same time as normal regeneration.

In the Nichols-Herreshoff Furnace which will be described shortly, the char is heated directly by the products of combustion of fuel oil or gas and hence it is a relatively easy matter to control the air supply

necessary for combustion in such a manner that the carbon content of the stock working char remains constant.

The method by which the char is drawn from the bottom of the cooler pipe has been a subject for much ingenious inventive ability and the basic problem is, in fact, a fairly complex one. The char has to be drawn off in such a manner that air is not admitted, that the char is not subjected to any crushing action, that the speed of draw-off may be easily varied, that the moving parts are not subjected to much wearing action, or particles of iron will find their way into the char, and that no lubrication is required.

Draw mechanisms have been studied and patented for nearly a hundred years and they include sliding plates, oscillating buckets, rotating buckets, gates and eccentrically rotating discs, and are illustrated in the accompanying diagram.



CHAR DRAW MECHANISMS

Fig. III

Theory and practice indicate that the row of kiln pipes nearest to the fire can regenerate the char more quickly than the pipes further back which are at a slightly lower temperature and thus the rate of draw-off should be adjusted to give the same effectiveness of regeneration throughout. Tests for the effectiveness of regeneration will be detailed later in this paper.

## (2) The Nichols-Herreshoff Furnace

The Herreshoff furnace was originally an ore roasting furnace and was in use in America at the end of last century, but its development for use as

a char regenerating furnace only dates from about 1938. It is illustrated in the accompanying diagram.

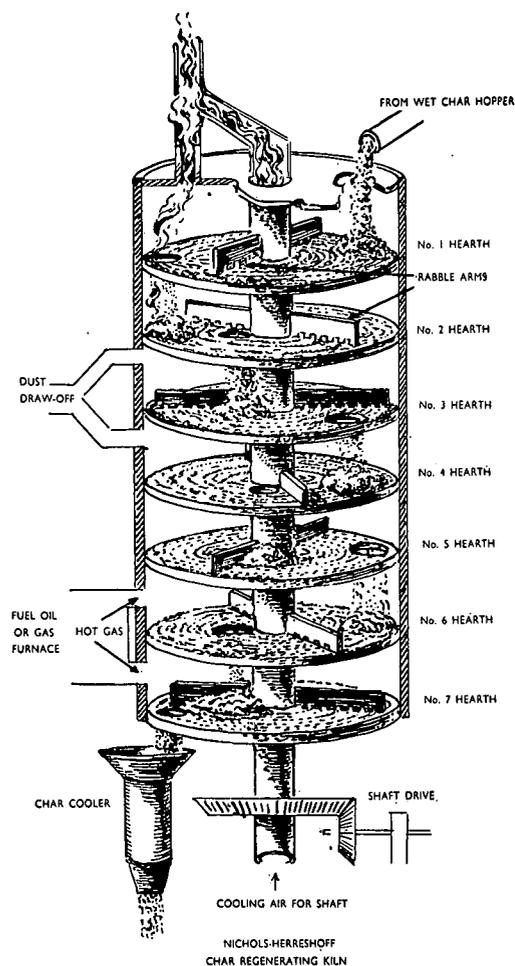


Fig. IV

The main disadvantage of the old Herreshoff was that the vertical driving shaft and the rabble arms had a very short life and this type of furnace only began to achieve success when these were air or water-cooled from the inside. One such design had a hollow driving shaft no less than 4 feet in diameter and the rabble arms could actually be withdrawn for replacement through this central shaft.

The development towards its use for char regenerating in sugar refining stemmed from the requirements in the oil refining industry to regenerate such adsorbents as Fuller's Earth, bauxite, special clays, silica gel and also bone char. Even as recently as 1930, the demand for bone char by oil refineries was as much as 15 per cent of the requirement for sugar refining and since this type of furnace was capable of regenerating bone char after the adsorption of oil impurities, it was natural that its use for regenerating bone char which had been used for sugar refining operations should be investigated.

The wet char falls into the uppermost hearth from the hopper and by the action of the rabble arms, it moves spirally outwards and eventually falls through openings at the outer edges of this hearth onto the hearth below. There it moves spirally inwards towards openings at the inner edges of this hearth.

The products of combustion of fuel oil or gas are passing upwards as a counter current flow and the air supply is controlled so that there is about 2 per cent excess oxygen which, for some reason, does not have any decarbonizing effect on the char.

As mentioned earlier, if there is a requirement to induce a little decarbonization, the air supply can be increased to allow sufficient excess oxygen to cause this reaction and since it has been found that 8-10 per cent oxygen is required to commence the decarbonization reaction, the volumes are not very critical.

In general, the through-put rate of a Nichols-Herreshoff is much greater than the rate for a Scotch Kiln, and it has been calculated for the Hulsar Refinery that one Nichols-Herreshoff of the same size as that installed at the Liverpool Refinery of Tate and Lyle would be sufficient to regenerate all the char that is required, whereas nineteen Scotch Kilns are at present installed.

The cheaper fuel oils contain a few per cent of sulphur and this can cause a rise in the sulphur content of the regenerated bone char, but if necessary other forms of liquid or gaseous fuel can be used if it is uneconomical to purchase fuel oil of less than 1 per cent sulphur content.

The firing is done into the lowest two, or the lowest two but one, hearths, this latter arrangement being considered preferable to prevent the hot gases from impinging directly into the cooler which would occur with the first arrangement if the flow of char was stopped for any reason.

The cooler comprises a series of vertical pipes around which water flows and this is designed to form part of the heat economy system of an efficient refinery. It is, in other words, a heat exchanger.

In a later modification at Liverpool, air is used instead of water and the heated air resulting is then forced through the incoming wet char in the uppermost hearth to improve the thermal efficiency of the Nichols-Herreshoff furnace.

## USE OF CHAR IN THE REFINERY

### (1) Dust and Heavy Particle Separation

After regeneration, the char is passed over a Kipp-Kelly Gravity Separator which comprises a vibrating perforated metal sheet table which has one or more raised riffles and through which, in an upward direction, a stream of air is blowing. The table is

slightly tilted and the air stream first of all lifts up the dust which is extracted through a hood and the particles of lighter density char are also raised, and they can then travel over the riffles. The particles of heavier density char are lifted progressively less as the density rises and these continue to travel along the surface of the table without passing over the riffles.

Thus the good char is separated from the dust which is not always wanted, since it necessitates, a greater pressure to force the brown liquor through the char cistern, and heavy density particles which are also not wanted, since their pores have become clogged with ash extracted from the brown liquor which has not been efficiently removed during the washing process. The dust and the heavy particles constitute only a small proportion of the char and the particles which have good colour and ash removal properties are thereby separated from char particles which are not of much use.

### (2) Filling of Cisterns

If the cistern is filled with dry char and then brown liquor is led into the top of the cistern, it would take a long time to penetrate and there would be many pockets of char which would probably never become wetted by this liquor.

Another reason why this method, known as "dry settling" is not used is that there is an exothermic physical reaction when char meets water. It will be remembered that when char is being dried, a considerable amount of heat is required to remove the adsorbed water, as distinct from the surface water, from the pores of the char, and thus heat is liberated when the reverse takes place and if the brown liquor already at about 150–160°F met the dry char at a temperature not lower than this, and slowly penetrated through a 20-foot depth, the leading liquor would quickly be raised to a temperature where colour formation or caramelization would take place.

Cisterns are usually prepared for use by "wet settling" which process is as follows: Over the false bottom of the cistern, the sheet of canvas is laid and a 4-foot layer of dry char is placed on the top of this. Care is taken to ensure that the char sheet fits snugly around the perimeter of the cistern, and this char is then covered with water. The water is allowed to drain out and brown liquor is led into the cistern until it is a little over half full. Dry char is now added through a spreader which is a rotating funnel with an offset lower tube designed to prevent segregation of char of different sizes in the cistern, and the amount of brown liquor which has previously been added should be calculated to be just sufficient to completely fill the cistern when it has been filled with char to within about a foot of the top.

The cistern lid is now bolted down and the brown liquor flow commences from a high level tank which gives about 30 feet of hydro-static head.

### (3) Sweetening off

After a predetermined number of hours running, generally in the region of 70–100 hours at a flow of about 500 gallons per hour, it is considered that the colour and ash removing properties have been reduced to an uneconomic level and the brown liquor flow is discontinued.

It is possible at this stage to use what is known as a two-liquor system in which the brown liquor flow is followed by a liquor from much darker sugars such as are produced in the refinery recovery department but at the moment, Hulsar Refinery uses a one-liquor system and thus when the brown liquor flow is discontinued, it is replaced by warm water. After a period of time which is dependent on many factors, the polarization of the issuing sweet water will fall to the figure of 0.13° at which point the Durban Corporation will allow the flow to go into the sewerage system. Everything that has come out of the cistern before this extremely low level of sugar contamination has been reached, has to be retained in the refinery and put to whatever use is most convenient, and all the water which has been used for sweetening-off eventually has to be evaporated.

### (4) Washing Off

The washing to drain is continued for 12–15 hours at an increased rate in order to wash out the adsorbed ash and since most of the ash is in the form of calcium salts, it has been found that water at about 140°F is more efficient in removing this ash than hot water. The presence of carbonate ions in the wash water has also been found to aid the desorption of calcium sulphate and calcium phosphate and thus it is the practice in some refineries to add a little soda ash to the wash water when washing to drain.

The water flow into the cistern is now stopped and the char is allowed to drain for several hours, after which time the man-hole, which is just at the false bottom level, is opened and the char is raked out on to a conveyor. From the conveyor, it travels *via* a bucket elevator to the hoppers above the char kilns, and the whole cycle is then complete.

## LABORATORY TESTING OF CHAR

Firstly there are the routine tests which are done to assess the regenerating efficiency and these are relatively simple.

100 grammes of char from the kiln are placed in a bottle and shaken up with 100 millilitres distilled water for 1 hour, and either after allowing the char

to settle, the supernatant layer is poured off, or if the char does not settle well, after centrifuging, the pH of the aqueous extract is determined. A well regenerated char will show a pH of 8.5 to 9.0 and if the pH is lower than this, it will generally be found that the kiln temperature has been allowed to drop below the usual operating temperature.

Another test which gives a good indication, but not a very accurate quantitative measure of the conditions to which the char has been subject during its flow through the kiln is the "Caustic Soda" test. 10 millilitres of char are placed in a boiling tube and 20 millilitres of 2 per cent caustic soda solution added. The test tube is placed in a bath at approximately 105°C and the mixture allowed to boil for two minutes. After cooling and settling, the colour of the caustic soda extract is matched against an arbitrary standard series. Then a few millilitres of lead acetate solution are added and the second colour also matched against the same series.

The theory behind this test is that if the char has been under-burned, some of the sugar remaining in the pores will not have been distilled off and will impart a colour to the caustic soda solution ranging from pale straw to a good brown colour according to the quantity. If the char has been over-burned, however, some of the adsorbed calcium sulphate will, in the presence of the carbon in the char, and at the regeneration temperature, have been reduced to calcium sulphide and this will give a colour with lead acetate solution, the depth of which colour, once again, giving an indication of the extent of the over-burning.

Frequently one finds both over-burned and under-burned char even from the same kiln pipe and this is explained by the difference in the maximum temperature attained by the char in contact with the kiln pipe walls from the char that travels throughout at the centre of the pipe.

### HULSAR BONE CHAR TESTING APPARATUS

The systematic checking of the colour and ash removing properties of the working stock char, as well as the competitive comparison of these properties for deliveries and sample offers of new char, is a more complicated procedure and it was for this purpose that the Hulsar Bone Char Testing Apparatus was planned, designed and constructed.

It will be seen from the diagram that the apparatus consists of an inner vessel acting as a supply tank for the brown liquor within a water bath which is kept at a constant temperature by means of water circulating from a thermostatically-controlled water supply vessel fitted with a centrifugal pump, and that the annular space between these two is fitted with eight tubes which take the place of the refinery char cisterns.

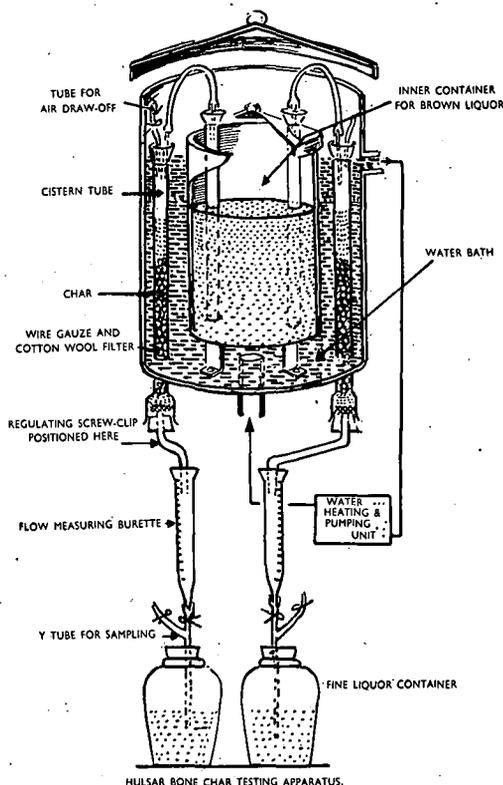


Fig. V

The incoming water enters at the centre of the base of the water bath and the inner vessel has a skirt around its lower edge allowing this water to flow outwards then upwards around the cistern tubes and finally through an overflow back to the circulating pump.

The cistern tubes have a  $\frac{1}{4}$  inch wad of cotton-wool sandwiched between two wire gauze circles above the single-holed neoprene stopper which is at the lower end, this being to prevent the char from flowing out whilst it does not hinder the liquor flow.

As is to be expected with a piece of apparatus which is not based on any conventional design for the reason that there is no conventional design for a bone char testing apparatus, a number of minor faults in design and difficulties in operating procedure have been shown up during preliminary decolourizing runs and the procedure for operating this apparatus is still in the experimental stage.

The tentative operating procedure is as follows: the water bath is raised to about 80°C by blowing in steam through a silent blower and then the heater/circulator is switched on to maintain this temperature.

Brown liquor from the refinery is heated to about 80°C and filled into the inner container and the lid to this container fitted. The wire gauze cotton-wool

sandwich is placed in position at the lower end of the cistern tubes, the lower bung is inserted tightly, and the rubber tubing leading away from this bung is closed with a pinch clip.

300 millilitres of 65° Brix refined sugar liquor are poured into the cistern tube and a wide-stemmed funnel inserted into the upper open end of the tube.

About 350 millilitres of dry bone char are now poured in at the rate of 12 millilitres every 10 minutes. 10 minutes after the final portion has been added, the depth of char is measured by a dipstick and the volume adjusted, if necessary, to give exactly 350 millilitres.

The upper two-holed bung is now fitted and the connections made to the tubes which lead through the lid of the inner container to the bottom of this vessel.

Air is now drawn out of the cistern tube by applying a vacuum to the other rubber tube which goes through this upper cistern tube bung and brown liquor flows up its tube inside the inner container and eventually it will be found that all the air has been withdrawn and thus the syphon is complete.

The pinch-clip on the tube connected to the lower cistern tube bung is now opened and the flow of liquor commences. By means of a regulating screw clip and a 25 millilitre burette which forms part of the liquor path, the rate of flow is adjusted to 30 millilitres per hour.

The refined sugar liquor must be withdrawn before the decolourizing run commences and this period of time enables the regulation of the flow rate to be completed.

The flow proceeds *via* this flow-measuring burette into 2-gallon stone jars and there are glass Y pieces just above the neck of these jars which enable samples to be withdrawn from the jar without stopping the flow through the Char, since the flow can accumulate in the flow-measuring burette whilst cumulative samples are being sucked out *via* the other arm of the Y piece.

Measurements are made periodically of the cumulative values of colour, ash and pH and the flow continues until an arbitrary value of colour has been exceeded. Colour values are plotted against time or volume and the exact time or volume at which this arbitrary colour standard was just reached can be determined.

Since it has been found that the colour removal and ash removal properties of bone char are, to a certain extent, mutually related and further that these properties vary with the actual levels of colour and ash in the brown liquor, it is necessary to adjust

the ash content and colour of the brown liquor to arbitrary standards before the run commences. The standards are not entirely arbitrary, either for brown liquor or fine liquor, since as far as possible, they are both related to the levels in use in the refinery, or to levels which we hope to attain in the near future.

The adjustment of colour and ash is done by the addition of refined sugar liquor to give a fixed ash content whilst a little variation is allowed in the colour value.

A point about which no great measure of agreement has been reached is the effect of the size of the bone char particles, beyond the obvious one, that the smaller the particle, the greater the total surface area and thus the greater will be the colour removal property, but even this generalization is complicated by the fact that the char particles are porous and the surface area of these pores is not increased by reduction in size, in fact, since breakage is probably more likely to occur along a pore, the pore area may actually be decreased by reduction in size of the particle.

We have decided that the easiest way out of this difficulty is to sieve a larger quantity of char than is required for the test, using about five or six sieves covering the range of the specification and then we take the same weight of each of the separate chars we are comparing from each of the sieved fractions and thus each char to be compared is reduced to a common sieve analysis, or in other words, it has a synthetically standardized grist.

To make this procedure a little clearer, we sieve through Tyler Sieves of 9, 10, 12, 20, 28 and 35 meshes and make the following separation: Particles retained on the 9-mesh are rejected, 100 grammes are taken from the particles on the 10-mesh, 100 grammes are taken from the particles on the 12-mesh, 150 grammes from the 20-mesh, 75 grammes from the 28-mesh and 75 grammes from the particles retained by the 35-mesh.

This gives 500 grammes total and after thorough mixing, this is the char used for filling the cistern tubes.

Thus the tests that we perform will not compare one char with another directly or as supplied by the manufacturer, but will merely act as a comparison between known weights of known size fractions of the two chars. Fortunately, most char manufacturers supply material of fairly comparable grist analyses and it may be found necessary for the refinery to specify to the manufacturers what grist analysis is required if their normal product does vary considerably from what is considered as satisfactory as a result of our laboratory testing.

When the decolourizing run has been concluded, the brown liquor supply is discontinued and it is replaced by warm water. The flow rate is increased and the sweetening-off and washing operations as described earlier for refinery practice are repeated on the laboratory apparatus.

The char is then transferred to an iron tube similar in shape and size to the cistern tubes and a laboratory scale regeneration performed. The kiln tube has a screwed cap which has a small screwed plug in it which closes up a very small hole. The tube containing the wet char is placed vertically in a furnace and the temperature raised to 105–110°C with this small valve open to allow steam to come out. After drying is complete, the temperature is raised to 450–500°C for about 4 hours with the small valve just open so that whilst decomposition products can get out, air cannot find easy ingress. At the end of four hours, the valve is closed tight and the furnace switched off. When all is cool, the cap is unscrewed and the char removed ready for a second decolourizing run.

At this point, a sieving analysis is done to see if the regeneration has caused any gross dust formation, which would be a point against the sample of char.

The usefulness of any char is considered as the volume of liquor of an acceptable cumulative colour which a certain weight of the char will pass, coupled with the manner in which this volume is maintained through continued regenerations, due notice being taken of any excessive formation of dust during regeneration.

### NEW THREATS TO BONE CHAR

No account of the use of bone char in sugar refining would be complete without a mention of Synthad, a product of Baugh & Sons, Philadelphia.

Synthad is the closest approach so far to a synthetic bone char and from the chemical and physical standpoint it is a very close match.

Being a synthetically made material, it has the one advantage that most synthetic materials have over their natural counterparts, and that is that there is generally some manner in which the properties of the finished product can be modified by some alteration in the process of manufacture.

In the case of Synthad, it is possible to alter the initial hardness of the material, or the manner in which this hardness is maintained, or is not maintained, during use, and also the porosity of the material and the crystalline characteristics can be altered and thus it is theoretically possible that eventually Synthad may replace bone char which is so dependant for its properties on the selection of

the bones used as starting material merely because of the uniformity of the product.

Up to three or four years ago, the main disadvantages of Synthad were:

- (1) It requires a higher regeneration temperature, sometimes as high as 600°C and this brings with itself difficulties in the increasing of the temperature of existing kilns, and also the greatly increased attention required to the air-tightness of kilns, since at 600°C decarbonization occurs much more readily than at 450°C.
- (2) The regeneration rate of the conventional kilns in use has to be reduced when regenerating Synthad in order that the caustic soda test shall not show too serious an under-burning.
- (3) When the regeneration temperature of Synthad is reduced to 540°C in order to maintain the carbon content, it is very difficult to maintain the pH of the fine liquor coming off Synthad at over 7.0.
- (4) The colour removal property of Synthad is not as good as that of bone char.

The advantages of Synthad are—

- (1) The material is harder than bone char and therefore less susceptible to breakage during regeneration and when breakage does occur, it is generally a surface phenomenon only and thus the breakage products are in the form of dust which is easier to remove than pieces which are merely a little smaller than the original.
- (2) The pores are not as susceptible to clogging as the pores of bone char and the carbon content originally at about 6–8 per cent can be reduced by allowing decarbonization to occur to a certain extent and then increased by regenerating with some sugar deliberately left on the Synthad with a consequent regaining of the original colour removal properties.

It will be recalled that if the carbon content of bone char is decreased and then increased in this manner, the additional carbon does not improve the colour-removing property. More recent work has been done on Synthad and is continuing at the present moment with large-scale trials in several of the American refineries and the point of main interest at the moment is that efforts are being made to reduce the pore volume of Synthad since it has been discovered that this was the main reason why a higher regeneration temperature was required. It was found out in tests only about two or three years ago that this larger pore volume in Synthad was retaining more water than in bone char and the higher temperature of regeneration was required to remove

this water. It would be fairly obvious that a very much slower draw-off rate would be the most sensible way of overcoming this difficulty and when this method was tried, the regeneration temperatures were not required to be as high as 600°C.

Unfortunately, however, the draw-off rate had to be so drastically reduced that one refinery found that it would need another 35 per cent added to its kiln capacity.

Another new material which is at present being investigated in America and also in the bone char testing apparatus at the Hulsar Refinery, is Pittsburg Activated Granular Carbon Type CAL, a product of Pittsburg Coke and Chemical Company, Pittsburg. This is formed by the action of super-heated steam on bituminous coal of selected composition and is a most unusual substance because even though it consists mainly of carbon and has a very high colour adsorptive property, it is regenerated in a rotary furnace at about 750°C in the presence of excess air: thus regeneration comprises an oxidation of adsorbed colour rather than a distillation, as is the case with bone char, but the high temperature and the presence of air does not have a very marked effect on the granular carbon itself. Actually about 5 per cent replacement is required for every regeneration but makers claim that cycles of up to 30 days can be achieved. This would give an annual replacement rate of 50 per cent compared with 25 per cent for bone char. It will be recalled that the normal length of a cycle for bone char is of the order of 70—100 hours.

This material, unfortunately, possesses negligible ash removal power but it could conceivably be used with bone char in order to free the fine liquor from the last traces of colour.

### CONCLUSION

No paper dealing with bone char could conclude more fittingly than by quoting verbatim from Oliver Lyle's book—*Technology for Sugar Refinery Workers*:

"Bone Charcoal, or 'Char', as we call it, is a sugar refining material quite unequalled by anything else. There are bleaching processes which remove, often temporarily, the colour from coloured solutions. There are vegetable carbons which remove the colour entirely. But there is no other material which removes the colour and removes quite a lot of ash as well which is moderate in price, which lasts for many years, and which stands up to rough treatment without rapid loss of efficiency."

I should like to thank the Management of Hulett's South African Refineries Limited for permission to publish this paper, and my colleagues for their assistance and criticisms.

### GENERAL ACKNOWLEDGMENT AND REFERENCES

Deitz, V. R. (1947): Preliminary Survey of Bone Char Revivification and Filtration. Bone Char Research Project at National Bureau of Standards, Washington, U.S.A.

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### SUMMARY

An introduction deals briefly with the use of bone char in sugar refining and then an account is given of the history of the manufacture of char. Regenerating kilns with detailed reference to the Scotch Kiln in use at Rosburgh and the Nichols-Herreshoff Kiln in use by Tate and Lyle at Liverpool are next dealt with and then a more detailed account of the use of char in the refinery is given. This incorporates sections on dust and heavy particle separation, the filling of cisterns, sweetening-off and washing-off. The paper next deals with the laboratory testing of char, including an account of the Hulsar Bone Char Testing Apparatus and concludes with brief accounts of Synthad and Type CAL Granular Activated Carbon which may be regarded as potential dangers to the continued use of bone char in sugar refining.

**Mr. Narbeth** question the statement that it was very difficult to maintain the pH of the fine liquor coming off Synthad at over 7.0.

**Mr. Hastilow** said that most of his remarks on Synthad were taken from the references given. It was possible that Synthad has been improved since the Proceedings of the Technical Session on Bone Char published in 1955.

**Dr. Dodds** enquired if Mr. Hastilow could tell him anything of the chemical composition and structure of Synthad.

**Mr. Hastilow** replied that chemically and physically Synthad and bone char were very similar. Synthad was produced by compounding calcium phosphate, or basic calcium phosphate with special clay binders and various types of organic material and then heating these in a retort to a fairly high temperature in the absence of air so that the clay binder would soften and cement the calcium phosphate particles together. The organic material would then carbonise and yield carbon dispersed throughout the bulk of the small particles. Large scale tests were being made with Synthad at certain refineries and he thought that it would not be long before we heard much more about this synthetic product.

**Dr. Van der Pol** asked Mr. Hastilow why he aimed at a certain volume of 350 ml. of bone char rather than a certain weight. He wanted to know if there were a method of determining the surface area of the char as this was most important in eliminating ash.

**Mr. Hastilow** replied that the one method most used was to estimate the amount of nitrogen which could be absorbed by the char at the temperature of liquid air. He was not sure of other methods, but he

understood that there was one which depended upon the absorption of radio-active material. As far as the question of volume rather than weight was concerned, they had found by experience that most chars after being reduced to the standard grist had the same density, so it was not necessary to go to the trouble of weighing the char.

**Mr. Rault** said that the better ash removal of bone char compared with activated vegetable carbons could be explained by the higher amount of bone char used.

**Mr. Hastilow** replied that as far as he was aware vegetable carbons had no ash removal property and

thus the quantities used had no bearing upon this contention.

**Dr. Dodds** commented that the Nichols-Herreschoff Furnace had been used almost exclusively for the burning of pyrites fines in sulphuric acid manufacture for many years because of its excellent performance.

**Mr. Hastilow** said he was sure that anybody starting up a new refinery would never put in an old-fashioned Scotch kiln, but go exclusively for the Nichols-Herreschoff Furnace. It was subject to many less troubles than the Scotch kiln and since there were no kiln pipes to deteriorate maintenance generally was much easier.