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# Sonolytic Removal of Sugar Colour in the Presence of Activated Charcoal

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## ABSTRACT

*Adsorption of sugar colour from the aqueous solution has been carried out on the surface of activated charcoal in the presence and absence of ultrasound. The characterisation has been done through X-ray diffraction (XRD), scanning electron microscopy (SEM) and FTIR spectroscopy. The percentage removal of sugar colour in the adsorbent load of 0.5 to 3.0 gm was 42.7 to 92.8 % in the absence of ultrasound (US) and 50.4 to 97.8 % in the presence of US. The effects of initial concentration of sugar, adsorbent dose and contact time on the decolourization of sugar colour have been examined under different experimental conditions. Mechanism of adsorption has been explained with the help of cavitation phenomena occurring in the process. Applied sonochemical method was found to be more effective in comparison to the conventional methods for sugar refining processes.*

***Keywords – Activated Charcoal, Adsorption, Decolourization, IU, Ultrasound.***

## 1. INTRODUCTION

The most important parameter for evaluation of sugar quality is the colour of sugar. The adsorption phenomenon has acquired many applications in technological and biological fields since its discovery. The physical chemistry of removal of colorants or even colloids involves an adsorption mechanism. Adsorption techniques employing solid sorbents are widely used to remove certain classes of colours found in sugar. However, amongst all the sorbent materials proposed, activated charcoal (AC) is the most popular for the removal of colour from aqueous sugar solution. The adsorption capacity of AC is mainly due to their structural characteristics and their porous texture which gives them a large surface area, and their chemical nature which can be easily modified by chemical treatment in order to increase their properties [1]. The efficiency of any adsorbent material to adsorb the desired compounds (colour, ash, colloids) depends on several factors, such as porosity, surface area, pore size distribution, bulk density, surface chemistry, hardness, pH, particle density, particle size, amount of water soluble minerals and its total ash content [2-7]. In the case of sugar decolourization; the decolourizing material should possess a pore size distribution favourable for adsorption of a mixture of poly dispersed constituents from highly concentrated sugar solutions. The activated charcoal has wide industrial applications. Among these are:

1. To remove of small concentration of products, this may have no colour, taste, or odour, but which cause poor crystal yield or poor crystal habit.
2. To remove of trace quantities of impurities having no original colour, but subject to oxidation or other changes this may develop colour at a later stage of processing.
3. To remove of impurities causing haze or turbidity, including those which do not precipitate out until after the product is sold.
4. To remove of trace quantities of water from water-immiscible organic solvents, or vice versa.
5. To remove the impurities from a liquid by extracting them with a trace quantity of immiscible solvent, and then adsorbing the resulting solution of solvent and impurities on carbon. This is useful where direct adsorption is unsuccessful [8-9]
6. To remove of trace quantities of ionic metals, by complexing with an adsorbable organic reagent and adsorbing the complex [10]

Ultrasonic is an effective purification and separation technique used in industry especially in wastewater treatment. Ultrasonic generates high-energy which causes the cavitation phenomenon which is the rapid and repeated formation and resulting implosion of micro bubbles in a liquid resulting in the propagation of microscopic shock waves. Microscopic vapor bubbles are created at site of rarefaction as the liquid fractures or tears because of the negative pressure of the sound wave in the liquid. Finally the compression part, which follows the rarefied part, collapses the bubble. The cavitational collapse of bubble generates localized hot spots of temperature as high as 5000K and pressure of as high as 1000 atmospheres for a life time of a few microseconds thus creating high energy movements of the solvent that results in localized high energy shear forces as shown in following Fig. 1 [11-12]. The number of cavitation bubbles collapsing per second may well be in the millions hence their cumulative effect can be significant. Shock waves from cavitation in liquid-solid slurries produce high velocity inter particle collisions, the impact of which is sufficient to convert the solid molecule into semi solid molecule. Thus the ultrasonic energy may be effective to remove the sorbed contaminants, like heavy metals, from fine grained soils.

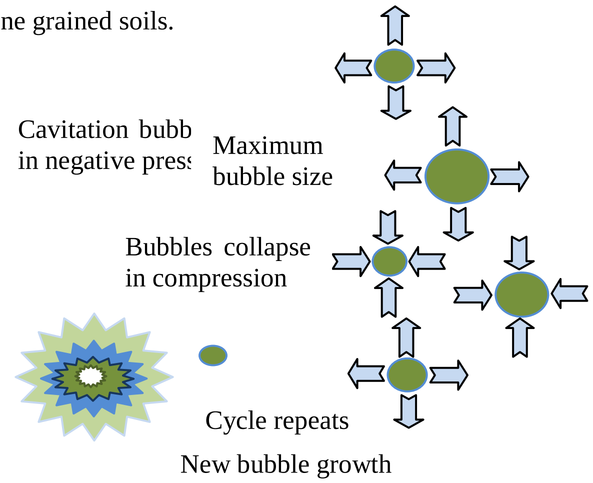

Figure 1: Cavitation and implosion phenomena in ultrasonic [12].

According to Fuchs 2002, in elastic media such as air and most solids, there is a continuous transition as a sound wave is transmitted [13]. In non-elastic media such as water and most liquids, there is continuous transition as long as the amplitude or "loudness" of the sound is relatively low. As amplitude is increased, however, the magnitude of the negative pressure in the areas of rarefaction eventually becomes sufficient to cause the liquid to fracture because of the negative pressure, causing a phenomenon known as cavitation. As the wave fronts pass, the cavitation bubbles oscillate under the influence of positive pressure, eventually growing to an unstable size. Finally, the violent collapse of the cavitation bubbles results in implosions, which cause shock waves to be radiated from the sites of the collapse. The collapse and implosion of myriad cavitation "bubbles" throughout an ultrasonically activated liquid result in the effect commonly associated with ultrasonic.

1.1 Advantages of using sonochemical method over other methods

There are immense advantages of using sonochemical method over other methods, as under:

1. Sonochemical method is a faster method, therefore more productive in terms of time and energy consumption [14-15].
2. Products synthesized by the sonochemical methods have thicker walls than the conventional methods, hence greater stability [16-17].
3. The control of particle size is easier in sonochemical process, which is otherwise not possible by any other technique [18-19].
4. Preparation of amorphous products is possible, therefore, enhanced characteristics such as adsorption, catalytic activity etc. may be expected [20-21].

According to Farid Chemat 2011 [22] the US technology has a significant effect on the rate of various processes in the food industry. Using ultrasound, full reproducible food processes can now be completed in seconds or minutes with high reproducibility, reducing the processing cost, simplifying manipulation and work-up, giving higher purity of the final product, eliminating post-treatment of waste water and consuming only a fraction of the time and energy normally needed for conventional processes. Several processes such as freezing, cutting, drying, tempering, bleaching, sterilization, and extraction have been applied efficiently in the food industry. The advantages of using ultrasound for food processing, includes: more effective mixing and micro-mixing, faster energy and mass transfer, reduced thermal and concentration gradients, reduced temperature, selective extraction, reduced equipment size, faster response to process extraction control, faster start-up, increased production, and elimination of process steps. Food processes performed under the action of ultrasound are believed to be affected in part by cavitation phenomena and mass transfer enhancement [23-25]. TABLE 1 shows the applications of ultrasound in food processing:-

Table 1. Applications of Ultrasound in Food Processing

| Applications | Conventional methods | Ultrasound principle | Advantages | Products |
|---|---|---|---|---|
| Cooking | Stove | Uniform heat transfer | Less time, Improving | Meat |

| | | | | |
|---|---|---|---|---|
| | Fryer<br>Water bath | | heat transfer and organoleptic quality | Vegetables |
| Freezing/ crystallization | Freezer<br>Freezing by immersion, by Contact. | Uniform heat transfer | Less time, Small crystals, Improving diffusion and<br>Rapid temperature decreasing | Meat<br>Vegetables<br>Fruits<br>Milk and products |
| Drying | Atomisation, Hot gas stream, Freezing and Pulverisation | Uniform heat transfer | Less time, Improving organoleptic quality and Improving heat transfer | Dehydrated products (fruits, vegetables) |

According to Suslick (1999) [26] the application of ultrasound in the synthesis of nanostructured materials, including both direct sonochemical reaction and ultrasonic spray pyrolysis method have great advantage. Ultrasonic irradiation provides unique energy is released to heat the contents of the bubble. These transient, localized hot spots with extremely high temperatures and pressures are primarily responsible for chemical effects of ultrasound. The usefulness of sonochemical synthesis as a synthetic tool resides in its versatility. With a simple modification in reaction conditions, various forms of nanostructured materials can be synthesized, including metals, alloys, oxides, sulfides, carbides, and nanos- tructured supported catalysts [27-29]. The sonochemical method has been even further extended to the polyymers, and biomaterials. Sonochemical decomposition of volatile organic precursors combined with significantly enhanced mass transport of materials via shock waves has been utilized in the synthesis of nanocomposites.

Aijun Hu (2006) reported [30] application of ultrasound on scale control in the Chinese sugar industry. According to him ultrasonic technique and equipment, the HTC and evaporation intensity of evaporators can be improved greatly, and the viscosity of the sugar solution decreases with ultrasound. The investigation results of evaporators indicated the HTC and evapo- ration intensity were improved by 42.4% and 15.2% respectively. The efficiency of removing scale ranged from 65% to 98%, and the average was 76.4% under ultrasonic treatment. With the treatment of ultrasound, the time of cleaning an evaporator was reduced to 38–75% of that without ultrasound. Singh (2013) described [31] the following important of the power ultrasound (PUS) in sugar industries:-

(a) Viscosity of massecuite and final molasses was reduced by 40-50 %.

(b) The physical characteristic of the scale was found to chance and favours the formation of vaterite which is thermodynamically least stable polymorphs of scale (calcium carbonate).

(c) Sodium hydroxide usages and disposal costs were reduced 65%.

(d) Hydrochloric acid usages and disposal were reduced 60%.

(e) Time to clean up the evaporators and labour cost is reduced significantly.

(f) Powder consumption was reduced.

## II. MATERIALS AND METHODS

The Activated Charcoal (reference carbon) was purchased from Sigma Chemicals. The brown colour sugar was collected from local sugar mill.

### 2.1 Fourier transforms infrared (FT-IR) spectroscopy

The AC (2 mg) was mixed with 200 mg of KBr and then pelleted. FT-IR spectra of the pellets were recorded using a Cary 630 FT-IR spectrophotometer.

### 2.2 Scanning electron microscopy (SEM)

The particle morphologies of the AC were studied using scanning electron microscope [SNE 3200 M (S. Korea)]. Samples were mounted on aluminum stab with the help of double-sided tape. Mounted stabs were coated with gold palladium prior to analysis using a Polaron sputter coater.

2.3 XRD (X- Ray Diffraction): XRD was done using Bruker AXS D8 advance (Germany).

### 2.4 Physical and chemical characteristics of activated charcoal (AC)

The physico-chemical properties such as apparent density, hardness, pH and mineral content of the Activated Charcoal were examined.

### 2.5 Methodology used for sugar solution colour measurement

The industry standard sugar solution colour measurement is the International Commission for Uniform Methods of Sugar Analysis (ICUMSA) colour method [32]. The absorbance of the test solutions were recorded using UV double beam spectrophotometer. The colours of the samples were determined using the ICUMSA protocol. ICUMSA colour is an indication of the overall colour of the sugar solution.

The colour is calculated as follows:

$$\text{I.U.} = \frac{Abs \times 1000}{b \times C}$$

## III. RESULTS AND DISCUSSION

### 3.1. Characterization of the materials

### 3.1.1. FT-IR Spectroscopy

Interaction of the adsorbent material with decolourization is explained through FTIR spectrum so obtained. Fig. 2 shows the FTIR spectra of blank AC, after sugar decolourization process in the absence of ultrasound and in the presence of ultrasound (US). Peaks around 1630 $cm^{-1}$ correspond to C=O group. After sugar decolourization adsorption, it is observed that there is an increase in the transmittance and also more peaks are introduced into the spectrum. The bands in the range of 3650 - 4000 $cm^{-1}$ have also been attributed to the hydrogen-bonded -OH group of alcohols and phenols (sugar colourant) [33-34]

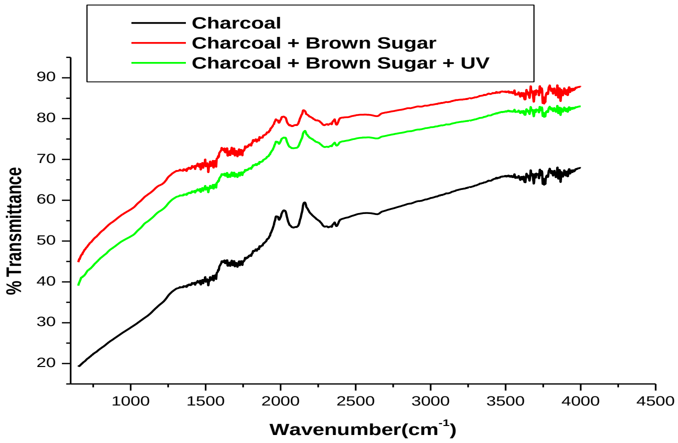

Figure 2: FTIR spectra of blank AC, after sugar decolourization in the absence of ultrasound and in the presence of ultrasound.

3.1.2 SEM Characterization

SEM images of AC before and after decolourization process in the absence of ultrasound and in the presence of ultrasound is shown in Fig. 3-5. The results show that there is uneven surface morphology with significant pores and fibrous structure. After adsorption, the pores get filled up as can be seen from the figure below.

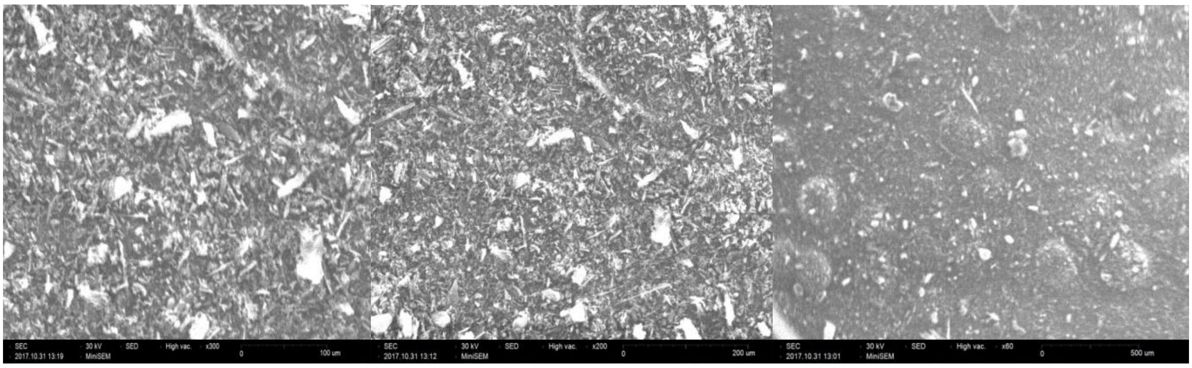

Figure 3: SEM pictures of AC before adsorption at different magnification.

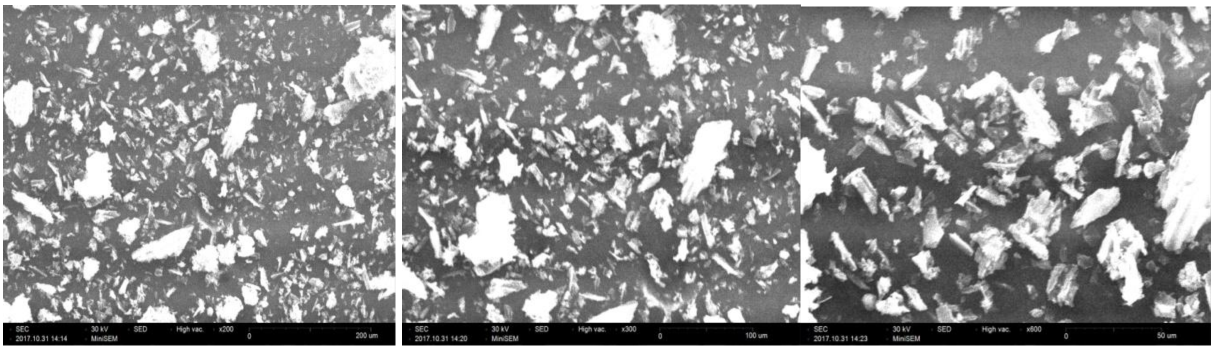

Figure 4: SEM pictures of AC after adsorption at different magnification without US.

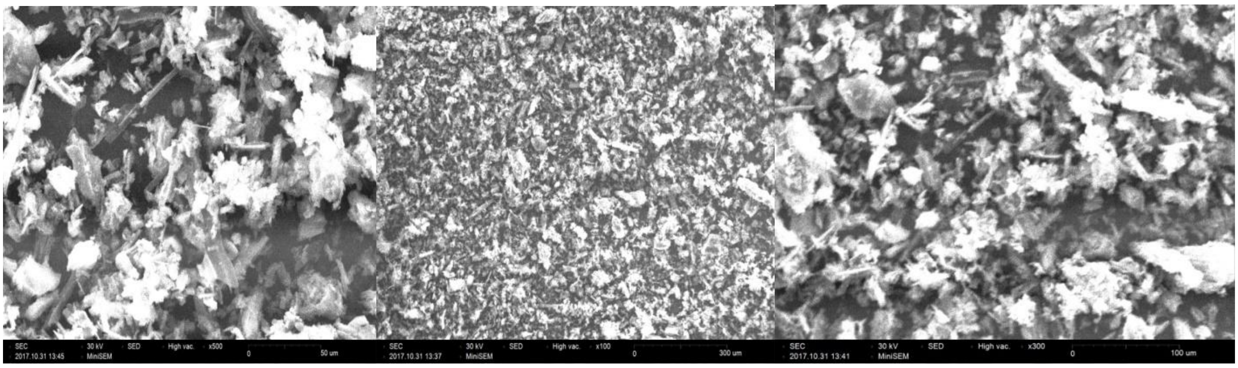

Figure 5: SEM pictures of AC after adsorption at different magnification with US.

### 3.1.3 XRD (X-ray Diffraction)

The X-ray diffraction technique was used in order to find out the nature of adsorbent material. The result obtained from XRD technique is presented in Fig. 6. It was observed that, the important diffraction peaks are 20.0°, 26.58° and 45° and phases corresponding to these peaks as Quartz and Cliftonite (C). Physical and Chemical composition of Activated Charcoal (AC) is tabulated in Table 2.

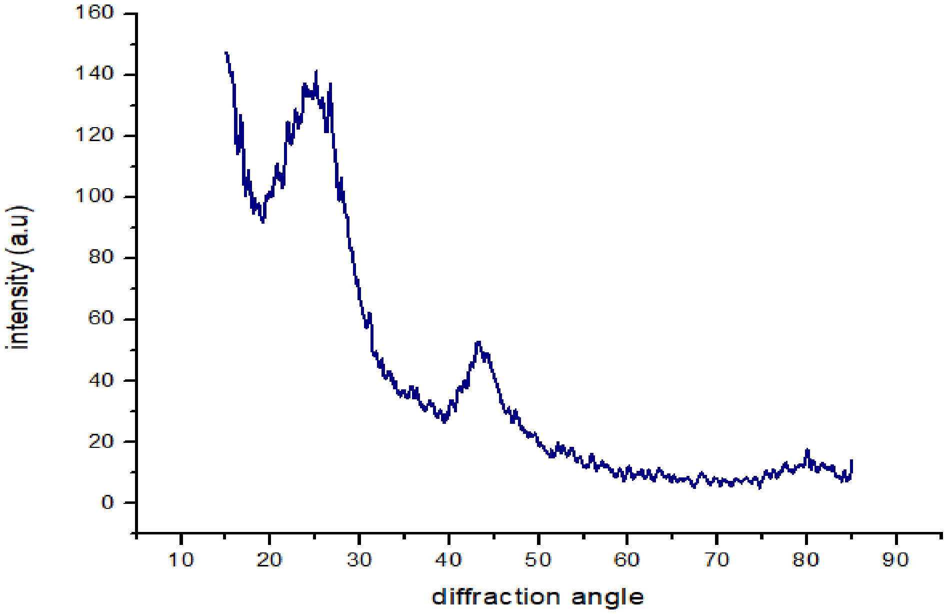

Figure. 6: XRD pattern of the AC.

Table 2. Physical and Chemical Properties of Activated Charcoal (AC) [35]

| Parameter | Technique Used | Measured Value |
|---|---|---|
| Carban Content | Carbon Analyzer | 93.60% |
| pH | pH Meter | 7.2 ± 0.04 |
| Conductivity | Conductivity Meter | 26 ± 2.5 |
| Bulk Density | Powder Density Meter | 0.25 g/ml |
| Specific Surface Area | $N_2$ Gas Adsorption | 1043 m2/g |
| Porosity | Hg Intrusion | 61.20% |
| Average Particle Size | Sub-Sieve Sizer | 4µ |
| Percent Iodine Removed (PIR) | Titration Method (Hassler, 1963) | 97 ± 2.0/0.5g |
| Percent Molasses Colour Removed (PMCR) | Titration Method (Hassler, 1963) | 95 ± 2.0/0.5g |
| Ash Content (% dry weight) | Residue Weight | 4.25 ± 0.1 |

### 3.2 Effect of contact time

The decolourization results using aqueous sugar solution of varying contact time 30, 45, 60 and 75 minutes are described in Table 3 (without ultrasound) and in Table 4 (with ultrasound) respectively. Fig. 7 shows the effect of contact time on sugar decolourization without ultrasound and with ultrasound. Maximum efficiency of the colour removal is found at 45 minutes.

Table 3. Data showing decolourization results of raw sugar (30 B0x) (without US)

| Particulars | Blank | Contact Time | | | |
|---|---|---|---|---|---|
| | (30 $B^o_x$) | 30 Min. | 45 Min. | 60 Min. | 75 Min. |
| Absorbance at 420 nm | 1.512 | 0.810 | 0.747 | 0.718 | 0.705 |
| Colour (I.U.) | 3532 | 1892 | 1745 | 1677 | 1647 |
| % Decolourization or % Removal of Colour | - | 46.4 | 50.6 | 52.5 | 53.4 |

Table 4. Data showing decolourization results of raw sugar (30 B0x) (with US)

| Particulars | Blank | Contact Time | | | |
|---|---|---|---|---|---|
| | (30 $B^o_x$) | 30 Min. | 45 Min. | 60 Min. | 75 Min. |
| Absorbance at 420 nm | 1.512 | 0.602 | 0.517 | 0.499 | 0.493 |
| Colour (I.U.) | 3532 | 1406 | 1208 | 1166 | 1152 |
| % Decolourization or % Removal of Colour | - | 60.2 | 65.8 | 67 | 67.4 |

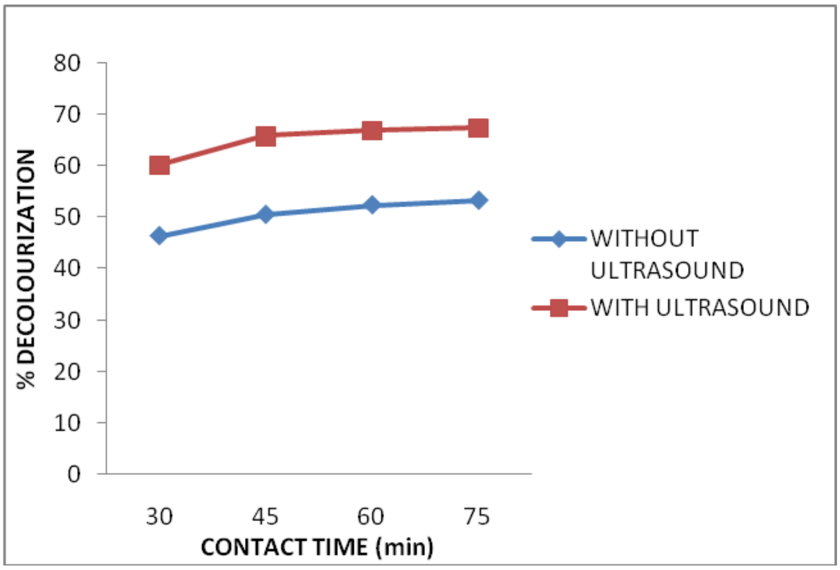

Figure 7: Effect of contact time on sugar decolourization without ultrasound and with ultrasound (Adsorbent dose-1 gm and sugar concentration- 30 $B^0{}_x$).

### 3.3 Effect of adsorbent dosage

The effect of various adsorbent dosages was studied at room temperature for 45 minutes contact time and 30 $B^0x$ sugar concentrations. The amount of adsorbent seems to influence the extent of uptake of sugar colour. Hence this factor needs to be taken into consideration in application of any adsorbent material. The maximum colour removal efficiency of AC is occurred at 1 to 2g. Further increase of adsorbent dosage did not cause any significant change in colour removal efficiency. It can be explained on the basis of attainment of equilibrium between the adsorbate and adsorbent at the existing operating condition rendering adsorbent incapable of further adsorption. The data included in Table 5 and 6 illustrate the colour removal efficiency with respect to adsorbent dosages. Fig. 8 shows the effect of adsorbent dosage on sugar decolourization.

Table 5. Data showing decolourization results of raw sugar (30 $B^0{}_x$) (without ultrasound)

| Particulars | Blank<br>(30 $B^o{}_x$) | Adsorbent Dosage | | | | | |
|---|---|---|---|---|---|---|---|
| | | 0.5g | 1.0g | 1.5g | 2.0g | 2.5g | 3.0g |
| Absorbance at 420 nm | 1.502 | 0.860 | 0.735 | 0.566 | 0.395 | 0.142 | 0.108 |
| Colour (I.U.) | 3509 | 2009 | 1717 | 1322 | 923 | 332 | 252 |
| % Decolourization<br>or<br>% Removal of Colour | - | 42.7 | 51 | 62.3 | 73.7 | 90.5 | 92.8 |

Table 6. Data showing decolourization results of raw sugar (300Bx) (with ultrasound)

| Particulars | Blank (30 $B^o{}_x$) | Adsorbent Dosage | | | | | |
|---|---|---|---|---|---|---|---|
| | | 0.5g | 1.0g | 1.5g | 2.0g | 2.5g | 3.0g |
| Absorbance at 420 nm | 1.502 | 0.744 | 0.521 | 0.390 | 0.214 | 0.108 | 0.033 |
| Colour (I.U.) | 3509 | 1738 | 1217 | 911 | 500 | 252 | 77 |
| % Decolourization<br>or<br>% Removal of Colour | - | 50.4 | 65.3 | 74 | 85.7 | 92.8 | 97.8 |

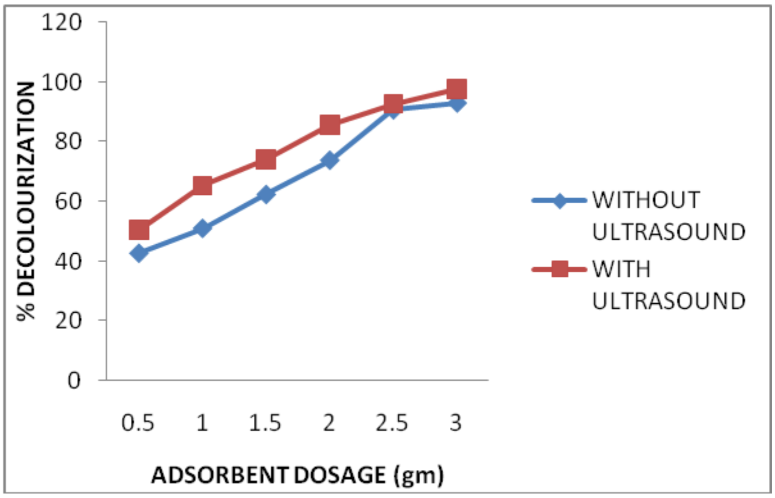

Figure 8: Effect of adsorbent dosage on sugar decolourization without ultrasound and with ultrasound.

### 3.4 Effect of sugar concentration

The effect of various sugar concentrations was studied at room temperature for 45 minutes contact time and one gram adsorbent dose. The maximum colour removal efficiency of AC is occurred at 25 $B^o{}_x$. Further increase of sugar concentration at constant adsorbent dose did not cause more significant change in colour removal efficiency. The data included in Table 7 and 8 illustrate the colour removal efficiency with respect to sugar concentration in the absence and in presence of ultrasound. Fig. 9 shows the effect of sugar concentration on sugar decolourization.

Table 7. Data Showing Effect of Sugar Concentration on Decolourization of Raw Sugar (without ultrasound)

| Particulars | Sugar Concentration (30 $B^o{}_x$) | | |
|---|---|---|---|
| | 25 | 30 | 35 |
| Absorbance of Blank at 420 | 1.250 | 1.510 | 1.758 |

| | | | |
|---|---|---|---|
| nm | | | |
| Colour of Blank (I.U.) | 3753 | 3528 | 3267 |
| Absorbance after adsorption at 420 nm | 0.492 | 0.743 | 0.864 |
| Colour after adsorption (I.U.) | 1477 | 1736 | 1606 |
| % Decolourization<br>or<br>% Removal of Colour | 60.6 | 50.7 | 50.8 |

Table 8: Data Showing Effect of Sugar Concentration on Decolourization of Raw Sugar (with ultrasound)

| Particulars | Sugar Concentration (30 B$^{o}_{x}$) | | |
|---|---|---|---|
| | 25 | 30 | 35 |
| Absorbance of Blank at 420 nm | 1.250 | 1.510 | 1.758 |
| Colour of Blank (I.U.) | 3753 | 3528 | 3267 |
| Absorbance after adsorption at 420 nm | 0.394 | 0.689 | 0.796 |
| Colour after adsorption (I.U.) | 1183 | 1609 | 1480 |
| % Decolourization<br>or<br>% Removal of Colour | 68.4 | 54.3 | 54.6 |

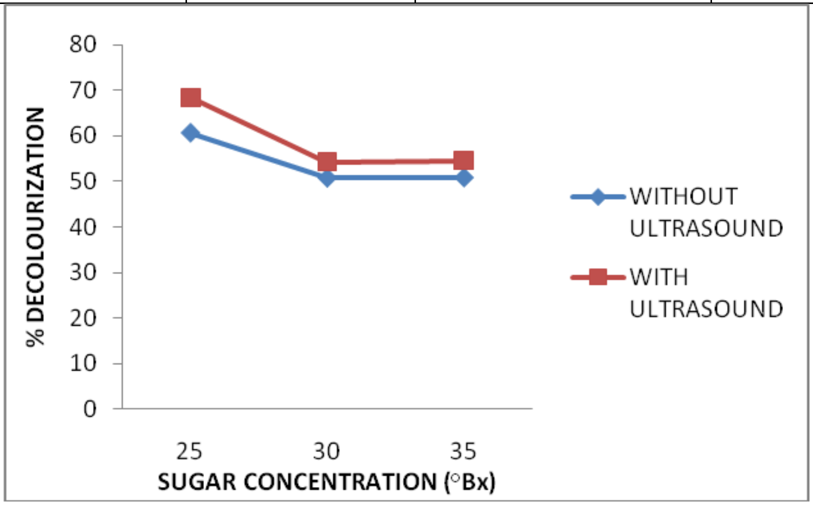

Figure 9: Effect of sugar concentration on sugar decolourization without ultrasound and with ultrasound.

### 3.5 Adsorption mechanism of sugar molecule on adsorbent

The separation technique based on the adsorption phenomena is based on the three distinct mechanisms: steric, equilibrium, and kinetic mechanisms. In the steric separation mechanisms, the porous solid has pores having dimension such that it allows small molecules to enter while excluding large molecules from entry. The equilibrium mechanism is based on the solid having different an ability to accommodate different species, that is the stronger adsorbing species is preferentially removed by the solid. The kinetic mechanism is based on the different rates of diffusion of different species into the pore; thus by controlling the time of exposure the faster diffusing species is preferentially removed by the solid [36].

According to Kamali (2010) the porous solid of a given adsorption process is a critical variable. The success or failure of the process depends on how the solid performs in both equilibrium and kinetics. A solid with good capacity but slow kinetics is not a good choice as it takes adsorbate molecules too long a time to reach the particle interior. This means long gas residence time in a column, hence low throughput. On the other hand, a solid with fast kinetics but low capacity is not good either as a large amount of solid is required for a given throughput. Thus, a good solid is the one that provides good adsorptive capacity as well as good kinetics.

To satisfy these two requirements, the following aspects must be satisfied:
a) The solid must have reasonably high surface area of micropore volume
b) The solid must have relatively large pore network for the transport of molecules to the interior

To satisfy the first requirement or statement, the porous solid must have small pore size with a reasonable porosity. This suggests that a good solid must have a combination of two pore ranges: the micropore range and the macropore range. The classification of pore size as recommended by IUPAC is often used to delineate the range pore size:

| | |
|---|---|
| Micropores | $d < 2$ nm |
| Mesopores | $2 < d < 50$ nm |
| Macropores | $d > 50$ nm |

The adsorption efficiency [37] of the sorbents are influenced by operating temperature, the chlorine or sulphur content in the feed waste, kinds of sorbents, sorbent size, amount of sorbent additive, and air flow rate. Among these factors, operating temperature, chlorine content, and sorbent size are most important. When heavy metals are heated, some volatilized metals exist in the gas phase, some form particles, and some are captured by sorbents through heterogeneous condensation and chemical adsorption. The third mechanism is particle capture. Most metal particle can be captured by sorbents through coagulation, coalescence, and scavenging. This is evident when the surface of sorbents is stickier, such as a glassy surface or sorbents coated with a layer of sticky material.

There are three general types of adsorption: physical, chemical, and exchange adsorption. Physical adsorption is relatively nonspecific and is due to the operation of weak forces of attraction or van der Waals forces between the molecules. The adsorbed molecule is not affixed to a particular site on the solid surface, but is free to move about over the surface. In addition, the adsorbed material may condense and form several superimposed layers on the surface of the adsorbent. Chemical adsorption, on the other hand, is the result of much stronger forces, comparable with those leading to the formation of chemical compounds.

## IV.CONCLUSION

After 45 minutes of ultrasonic treatment, the percentage removal of sugar colour in the adsorbent load of 0.5 to 3.0gm is 42.7 to 92.8 percent in the absence of US and 50.4 to 97.8 percent in the presence of US. The removal of sugar colour was significantly enhanced in the presence of ultrasound with adsorbents and the decolourization improved with increase in amount of adsorbents, under experimental conditions due to increased sorbent surface area and availability of more sorption sites resulting from the increased dose of the adsorbents from 0.5 gm to 3.0 gm. Ultrasound also improve the quality of sugar due to increase the colour removal capacity. The ultrasonic equipment is easy to operate, needs little maintenance and has a good automised performance. Ultrasonic technique or Sonochemical method is faster method, therefore more productive in terms of time and energy consumption in sugar refining processes.

## V.ACKNOWLEDGEMENT

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