

# **RESEARCH PAPER**

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# Assessment of Physico-Chemical Parameters of Sugar industry effluents

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## Abstract

Sugar industries wastewater effluents are responsible for significant environmental and health problems in the absence of adequate pollution control measures. Nearly all stages of sugar production results in discharge of wastewater containing high levels of oil and suspended chemicals. In the present study, water effluents were collected from selected sugar mills in Khyber Pakhtunkhwa (KPK) and the effluent samples were examined for different physico-chemical parameters *viz*. pH, total suspended solids (TSS), total dissolved solids (TDS), Electrical conductivity (EC), Turbidity, Free carbon dioxide, Carbonate, Bi-carbonate, Chlorides, Total hardness, Calcium carbonate, Calcium, Magnesium, Nitrite-nitrogen, Nitrate-nitrogen, Orthophosphate, and Sulfate. The analytical data revealed that the observed values of TSS, EC, TDS and pH were higher than Pakistan's National Environmental Quality Standards (NEQS) while other parameters turbidity, Calcium hardness, Nitrate-nitrogen, Nitrite-nitrogen, electrical conductivity, free carbon dioxide, and Sulfate concentration were found in range. The current study suggested that proper waste water monitoring and water arrangement system should be installed which not only ensure safety but also protect human health.

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#### Introduction

Water resource management has taken place all over the world in recent decades. Protecting the surface water resources from wastewater pollution plays a vital role for the sustainable water supply and development. The disposal of wastewater into the surface water bodies leads to serious problems and affects the people in health aspects (Goel, 2006; Azizullah *et al.*, 2011). Especially in the urban areas, domestic effluents and pollutants that discharges into the nearby surface water bodies created problems for the public (Barman *et al.*, 2000; Kisku *et al.*, 2000; Amathussalam, 2002). Hence, the effluent discharge affects the surface water bodies and quality which can cause many health problems to the public (Kanu *et al.*, 2011, ETPI, 2003).

The sugar production process is known to produce substantial levels of solid waste, waste water and noise pollution. The highly polluted wastewater from sugar mills, in particular, poses a substantial danger to human health and environmental quality (Adekunle, 2008). Several chemicals are used in sugar industries mainly for coagulation of impurities and of end products. In the past, sugar industries produced only sugar but nowadays these industries are involved in the production of sugar, electricity and ethanol (Basso et al., 2011; Pellegrini et al., 2011; Macrelli et al., 2012). Sugarcane is a valuable crop for bio-products because it produces sugar which has very high demand in the market and also bagass which provides energy in the form of fuel for the generation of electricity and steam (Chauhan et al., 2011; Renó et al., 2014; Eggleston et al., 2015). A significantly large volume of waste is generated during the manufacture of sugar and contains a high amount of pollution load, particularly in terms of suspended solids, organic matter, and press mud, bagasse and air pollutants (Gunkel et al., 2007; Abbasi et al., 2010; Muthusamy et al., 2012).

Pakistan's sugar industry, considered to be one of the best organized industrial sectors in the country, is also among the country's leading economic enterprises, directly or indirectly employing large number of people. Pakistan sugar industry comprises of 77 mills, 38 in the province of Punjab, 32 in Sindh, 6 in the Khyber Pakhtunkhwa (KPK) and one in Azad Jammu and Kashmir (AJK). The industry produced nearly four million tons of sugar during 2003-04 and has a full production capacity estimated at five million tones, well exceeding the estimated domestic demand of 3.6 million tons (GOP 2003). Wastewater from sugar mills with its high pH rapidly depletes the available oxygen supply when discharged into water bodies endangering fish and other aquatic life. Total Dissolved Solids (TDS) refers to all dissolved materials present in the water. Discharge of water with a high TDS level would have adverse impacts on aquatic life, render the receiving water unfit for drinking, reduce crop yields if used for irrigation and exacerbate corrosion in water systems and pipes (ETPI, 2003; Gupta et al., 2002, 2003). In the present study, an attempt has been made to investigate the waste water generated from the sugar mill in northern Pakistan and investigated the quality of wastewater in selected sugar mill effluents; characterize some of the physio-chemical water quality parameters in Mardan Sugar Industry. Chemical analysis of effluent samples from a number of sugar mills located in Mardan revealed the presence of several water pollutants in amounts exceeding National Environmental Quality Standards (GOP, 2003).Further, we compared the findings with NEQS and recommended some suggestions for policy makers.

#### Materials and methods

#### Study area and sample collection

Waste water samples were collected from selected localities of Mardanincluding Irum colony, Sharif Abad, Rustam colony and starting point of sugar mill and analyzed for different water quality parameters such as pH, electrical conductivity, turbidity, hardness, total dissolved solids (TDS), chloride, bicarbonates, carbonates, calcium, magnesium, sulfates, nitrate-nitrogen, Calcium carbonate hardness, Total hardness, orthophosphate and nitrite-nitrogen (Saranraj and Stella, 2014).

Sampling sites locations are summarized in Table 1. The wastewater is generally discharges to the surface water. Each sample was taken from four different points in every week in the sampling locations. Samples were collected in polythene bottles and analyzed in National Institute of Bioremediation lab Islamabad. Water samples were collected in s bottles and labeled (sampling point, date and flushing time). Sample collection was done with great care by holding the sample bottle cap in one hand while sample was collected in polyethylene bottles to to avoid contamination. Waste water samples were stored waste samples in ice-boxes with freezer packs and delivered to laboratory on the same day.

#### Determination of pH

Samples were taken in three beakers using the electrode of pH meter (LA, Lutron WA-2015) to determine the values. Spectrophotometric, depending on the equipment and light source used the pH value was estimated by means of a visual comparison of the color against a color scale and noted down once the pH was stable.

#### Determination of TDS

Determination of TDS was carried out by immersing the meter probe in sample or by using the electrode LA Lutron WA-2015. During this process stirred the sample vigorously using clean glass stirring rod. Then the observed reading was noted.

#### Determination of turbidity

Samples were taken in a beaker 25 mL and analyzed in viel/cuvette of turbidity. Place the vial/cuvette in turbidity meter and then note down the values of waste water sugar mill samples for turbidity.

#### Determination of free carbon-dioxide

Taking 25 mL of water sample in a beaker and then nearly adding 2 or 3 drops of phenolpthien indicator and stirring the samples. Then sample was titrated using standard NAOH (0.02N) a slight pink colour was appeared. The end point was recorded of the burette.

Free CO2 mgl<sup>-1</sup> = 
$$\frac{mloftitrantused}{volumeofsample} \times 1000$$
 Eq.1

#### Determination of hardness

25 mL of waste water sample was taken in a titration flask by means of pipette. 1mL ammonia buffer was added in sample. Indicator EBT (Eriochrome black T) was added in a sample. EDTA solution was taken in a burette. The initial reading was then noted. Samples were titrated against EDTA until the color changed. The final reading was noted.

mL of EDTA used = Initial reading-Final reading

Hardness = 
$$\frac{\text{ml of EDTA}}{\text{ml of sample}} \times 1000$$
 Eq.2

## Determination of Calcium

Sample was taken in a titration flask by means of pipette. 1mL Sodium hydroxide (ml solution) and Mouroxide ( $C_8H_8N_6O_6$ ) indicator capable of producing an unambiguous color change in a beaker. EDTA 0.01 M solution was taken in a burette. The initial reading was then noted down. Samples were titrated against EDTA until the color changed.. The final reading was noted.

ml of EDTA used = Initial reading-Final reading
$$Ca^{++} = \frac{ml \text{ of EDTA} \times 400.5 \times 1.05}{ml \text{ of sample}} \qquad \qquad \text{Eq.3}$$

Determination of Magnesium  $Mg++ = (A - B) \times 0.244$ Where A = Total hardness $B = \frac{ml of titrant used for calcium \times 1000 \times 1.05}{ml of sample}$ 

After calculating the value of A and B put these values in equation 1 and calculate value of magnesium.

Eq.4

#### Determination of Carbonates

Sample was taken in a titration flask by means of pipette. Indicator (phenephthelene) was added in a sample. Took Sulphuric acid solution in burette. The initial reading was then noted down. Samples was titrated against the solution until the color changed. The final reading was then recorded.

ml of H<sub>2</sub>SO<sub>4</sub> used = Initial reading-Final reading  

$$CO_{3}^{-} = \frac{ml \text{ of } H2So_{4}}{ml \text{ of sample}} \times 1000$$
 Eq.5

#### Determination of Bicarbonates

25 mL sample was taken in a titration flask by means of pipette. Added indicator methyl orange (2-3 drops) in a sample. Then sulfuric acid solution was taken in a burette and initial reading was then noted down. Samples were titrated against the solution until the color changed. The final reading was then noted down.

ml of H<sub>2</sub>SO<sub>4</sub> used = Initial reading-Final reading

$$HCO_{3^{-}} = \frac{\text{ml of H2So4}}{\text{ml of sample}} \times 1000$$
 Eq.6

## Determination of Chlorides

Sample was taken in a titration flask by means of pipette. Add 2-3 drops of indicator potassium chromate in a sample. Silver nitrate solution was taken in a burette. The initial reading was recorded. Samples was titrated against Silver nitrate solution until the color changed. The final reading was then noted down.

mL of AgNO<sub>3</sub> used = Initial reading - Final reading  

$$CO_3^- = \frac{\text{ml of AgNO3 \times 0.014 \times 35.5}}{\text{ml of sample}} \times 1000$$
 Eq.7

#### Determination of sulfates

Distilled water and 25mL of sample were taken in two beakers by means of pipette. Add 5mL of NaCl-HCl both in distilled water and sample. Then add a pinch of Barium chloride in both samples. Took two cuvettes and washed with distilled water. After that added distill water (prepared) sample into both cuvettes. Place the distilled water samples in spectrophotometer. Range of spectrophotometer for sulfates was 420 wavelength of visible range. Then set the reading of spectrophotometer at zero. Took out one cuvette of distilled water sample from spectrophotometer and left the other one as reference sample. Now took a cuvette and filled it with drinking water sample and placed it in spectrophotometer. Now note down the reading of absorbance for sulfates. Calculation was done by drawing a standard curve.

#### Determination of nitrite nitrogen

Distilled water was taken in two beakers by means of pipette. Added 1mL sulfanilamide nitrites in both distilled water and drinking water sample. Then add a pinch of NED (Naphthalene ethylene diamine) in both samples. Place the prepared samples for few minutes. Took two cuvettes and washed with distilled water. After that added distill water (prepared) sample into both cuvettes. Placed the distill water samples in spectrophotometer.

The range of spectrophotometer for sulfates was set at 543 wavelength of visible range. Now set the reading of spectrophotometer at zero. Then took out one cuvette of distill water sample from spectrophotometer and left the other one as reference sample. Then took a cuvette and fill it with drinking water sample and place it in spectrophotometer. The reading then was noted down. Calculation was done by drawing a standard curve.

#### Determination of nitrate-nitrogen

Distilled water was taken in two beakers by means of pipette. Added 0.5mL phenol disulphonic acid follow by 1mL of 12N KOH and then add 5mL of distilled water in both distilled water and waste water sample a yellow color will developed.

Then took two cuvettes and washed them with distilled water. After that added distilled water (prepared) sample into both cuvettes. Placed the distilled water samples in spectrophotometer. Setted range of spectrophotometer for sulfates was 410 wavelength of UV rang.

Then set the reading of spectrophotometer at zero. Now took out one cuvette of distill water sample from spectrophotometer and left the other one as reference sample. Took a cuvette and filled it with waste water sample and placed it in spectrophotometer. Now note down the reading of absorbance for nitrates. Calculation was done by drawing a standard curve.

#### Determination of Orthophosphate

Solution approximately 25mL was taken in a conical flask. Also run distilled water blank simultaneously. Now add 1mL ammonium molybdate solution and 3 drops of stannous chloride solution and shake the solution blue colour appear and wait for 10 minutes and now take a cuvette and fill it with waste water sample and place it in spectrophotometer at 690 absorbance. The reading of absorbance was recorded. Calculation was done by drawing a standard curve.

## **Results and discussion**

#### pH

The variation in the mean pH values in all the four sampling points have been explained in Table 2. pH of premier sugar mill wastewater was found within the permissible limit (6-9). However, at sampling point quite low pH value was recorded as compared to other points.

<b>Table 1.</b> Location and geographical coordinates of wastewater sampling point	Table 1.	Location and	l geographical	coordinates of	of wastewater	sampling po	oints.
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S. No	Location/Sample code	Geological site through GIS
1	Starting point	34.17129893 N,
	Sample # 1	72.031677 E
2	Sharif Abad	72.03334199995 N
	Sample # 2	34.1652947 E,
3	Rustam colony	34.167144 N,
	Sample # 3	72.029510 E
4	Irum colony	34.2781232 N,
	Sample # 4	72.1193567 E

## Electrical conductivity

The graphical representation shows the mean EC values in four sampling points in Figure 2. The lowest value of 0.741µSwas observed at sample No.3 while the highest value of 0.973µSwas recorded at sample

o1 location Quite low EC values were recorded in all the sampling points and no National Environmental Quality Standards guideline is available for Electrical Conductivity.

Table 2. Results of Physico-chemical analysis of Sugar mill wastewater.

Parameters	Starting point	Sharif Abad	Rustam colony	Irum colony	NEQS for wastewater
	Sample#1	Sample#2	Sample#3	Sample#4	quality EPA
pH	7.24	7.05	6.82	6.92	6-9
EC µS	0.973	0.918	0.741	0.810	Nil
TDS (mg/L)	660	621	492	533	3500
Turbidity	23.73	10.70	21.07	27.08	300
Free carbon dioxide (mg/L)	156	142	182	132	0.5 to 28.6
Carbonate (mg/L)	Nil	Nil	Nil	Nil	Nil
Bi-carbonate	56	50	58	142	400
Total hardness (mg/L)	296(25ml)	232	260	228	440
Calcium carbonate (mg/L)	180.6	142.8	96.6	88.2	200
Calcium (mg/L)	72.3303	57.1914	38.6883	35.3241	1000
Magnesium (mg/L)	28.1576	21.7648	33.16	34.112	150
Sulfate concentration (mg/L)	-	41.72727	5.909091	16.27273	600
Chlorides (mg/L)	-	55.664	404.2267	202.113(30ml)	1000
Nitrate-nitrogen concentration	6	3.334	5.112	4.112	30
(mg/L)					

#### Total dissolved solids

The graphical representation of wastewater of total dissolved solids by premier sugar mill is shown in Figure 3. The permissible limit for waste water to land water is 3500 mg/L but during sample analysis the total dissolved solids values were more than NEQS. The range of TDS was range from 492 mg/L to 660 mg/L.

## Turbidity

Organisms like phytoplankton can contribute to turbidity in open water. Erosion and effluent contribute to the turbidity of waste water, the light detector is at an angle of  $90^{\circ}$  to the light source the meter is considered a nephelometer, if it is at a  $180^{\circ}$ angle then it is a turbidity meter.



Fig. 1. Mean pH values at different sampling points.



Fig. 2. Mean electrical conductivity in four sampling point.

Turbidity can provide food and shelter for pathogens. The NEQS for turbidity is 300 (mg/l) and the wastewater turbidity was in permissible limit while the highest turbidity was found at sample location 04 with 27.08 turbidity. The turbidity values have been explained in Table 2.

#### Hardness

Total hardness, calcium, calcium carbonate and magnesium of waste water of sugar mill explained in Table 2. The NEQS for waste water quality is 440 mg/L and the total hardness of waste water is in permissible limit. Our sample hardness range from 228 mg/L to 296 mg/L.

## Calcium

The Graphical representation of Premier sugar mill waste water is summarized in Table 2. Experiments with sodium Oleate with different settings did not produce any significant recoveries, calcium affects capital and operation costs for the plant as well as the impact of ultimate sludge disposal on the environment. The calcium hardness for base point is more than other four points. The highest observed value for calcium among the sample was 72.33 mg/L. The NEQS of Pakistan for Calcium is 1000 (mg/L) so the effluent was in permissible limits.



Fig. 3. Mean TDS values in all the four sampling points.



Fig. 4. Mean turbidity values in all the four sampling points.

#### Magnesium

Magnesium is often precipitates in anaerobic reactors treating wastewater with high calcium content. Magnesium has the ability to disperse soil, when present above a certain threshold value. The graph of magnesium shows different values (Table 2). The values of magnesium in wastewater of Irum colony and Rustam colony was higher but in permissible limit. The NEQS for magnesium is 150 (mg/L) while the sample values range from 21.76 to 34.11 mg/L.



Fig. 5. Mean total hardness values in all the four sampling points.



Fig. 6. Mean calcium values in the four sampling points.

#### Calcium Carbonate

Sulfate and chloride compounds cause permanent hardness of water and hardness can be reduced by the addition of lime (calcium hydroxide).

The graphical representation of Premier sugar mill waste water has been represented by Table 2. The value of starting point or base point from where I collect the samples the value is very high from three other different locations all four values are in limit. The NEQS for calcium hardness is 200 (mg/L). *Bi-Carbonate, Chlorides and Free carbon dioxide* The bi-carbonate in the waste water samples was high but in permissible limit (Figure 9). The National Environmental Quality standard for bicarbonates is 400 (mg/L). Whereas Chlorides was found in permissible limits (Figure 10). The National Environmental Quality Standards for waste water effluent to land water is 1000 mg/L. The concentration of free carbon dioxide in the waste water is very high and it can affect the other water resources (Table 2).



Fig. 7. Mean Mg values in all four sampling points.



Fig. 8. Mean carbonate hardness valus in all four sampling points.

The NEQS for sugar industry is not available in the literature.

#### Nitrate-nitrogen concentration

The concentration of nitrates is commonly expressed as  $No_{3}$ - this nomenclature is used to differentiate nitrate nitrogen from nitrogen in the form of ammonia. Concentrations range from 0.1 mg/L to 3 or 4 mg/L in most areas. The graphical representation of nitrate-nitrogen concentration waste water has been explained in Table 2. The NEQs of nitratenitrogen concentration for sugar mill industry is 30 (mg/L).

## Sulfate concentration

Sulfate is one of the major dissolved components in waste water.

High concentrations of sulfate in waste water can have a laxative effect when combined with calcium and magnesium, the two most common constituents of hardness. Sulfate concentration of premier sugar mill waste water has been presented in Table 2.

The NEQs for sugar industry in Pakistan is 600 (mg/L). The concentration of Sulfate at Sharif Abad location is high but in permissible limits.



Fig. 9. Mean values chloride values in the four sampling points.



Fig. 10. Mean free Carbon dioxide level at four sampling points.

## Discussion

According to National Environmental Quality Standards of Pakistan existing standards for available parameters the allowed range of pH is one of the very important tool in the aquatic system and its indicator for the sustainability for the aquatic organisms. H The Electrical Conductivity also changes with alteration in the pH, EC, Total dissolve solids, turbidity, hardness, calcium, magnesium, Chlorides, carbonates, bicarbonates, total hardness. Calcium carbonate hardness, Magnesium, Calcium, sulfates, nitrates, Turbidity and nitrites 6-9, 3500 (mg/L), 1000 (mg/L), 300 (mg/L), 440 (mg/L) 150 (mg/L) and 600 (mg/L) respectively and some National Environmental standards are not found in literature.

The results achieved after the analysis of samples for pH value, which are within the permissible value but TDS are not in permissible limit.

The wastewater treatment plant has a high potential of removing key pollutants and could be used for better treatment of wastewater if managed properly.

The wastewater treatment plant is efficient; however parameters such as TSS and turbidity were unsatisfactory. pH values range from 6.5 to 7.5. Most of bio-chemical and chemical reactions are influenced by the pH.



Fig. 11. Mean value of nitrate-nitrogen at four sampling points.



Fig. 12. Mean sulphate values Sulphate concentration.

The reduced rate of photosynthetic activities reduces the assimilation of carbon dioxide and bicarbonates which are ultimately responsible for increase in pH. The low oxygen values coincided with high temperature during the summer month. The higher pH values observed suggest that carbon dioxide, carbonate-bicarbonate equilibrium is affected more due to change in physico-chemical condition. The value of free CO<sub>2</sub> ranges from 0.5 mg/l to28.6 mg/L. The maximum value (18 mg/L) was recorded in the month of July. This may be depends upon alkalinity and hardness of water body. The total dissolved solids fluctuate from 480 mg/L to 660 mg/L, the maximum value (356.4 mg/L) was recorded in the month of June. It is due to heavy rainfall. The turbidity of water fluctuates from 10.90 to 28.0 NTU. The maximum value of 27.08 NTU was recorded in the month of July; it may be due to human activities, decrease in the water level and presence of suspended particulate matter.

#### Conclusion

The management of waste water quality in premier sugar mill is increasingly a complex and multisectorial problem therefore few management activities should be done such as a slow sand filter may be introduced after the sequential batch reactor to improve the effluent wastewater quality of sugar mill, requires the ability to predict environmental consequences of the effluents and analyze remedial options both at the farm level because this waste water is then used for agriculture and it also contaminates the ground level drinking water.

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