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Chemical analysis in an effluent desalination system

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ABSTRACT

Due to rapid industrial growth and increase in population in developing countries, people of such places are facing severe water scarcity. They are also suffering from many water borne diseases, because of the unavailability of treated drinking water. Industries too face lot of problems to properly dispose the effluents. In this work, an attempt is made to solve the problems associated with effluent disposal and water scarcity in industrial areas. The effluent and desalinated water samples are tested at Tamil Nadu Water Supply and Drainage (TWAD) board. It is found that, the minerals present in the desalinated water sample are very low. As per the guidelines given by the TWAD board some minerals are added to make the water potable. The cost of the minerals to be added per liter of water is found to be 28 paise ($0.0056).

Key words: Solar desalination; Chemical analysis; Effluent treatment

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1. Introduction

Fresh water is one of earth's most valuable renewable resources. It is the essence of life and is a basic human requirement for domestic, industrial and agricultural purposes. Water is not only indispensable to industrial development, economic growth and social well-being, but also indispensable for the preservation of natural resources. For a country, a region or a civilization, securing an adequate water supply, has always been one of the essential prerequisites, not only to its development; but to its survival.

Nature, a very large-scale process of solar distillation, provides most of the required fresh water, through hydrological cycle. The importance of supplying potable water can hardly be overstressed. More than two-third of the earth's surface is covered with water. Most of the available water is either present as seawater or icebergs in the polar regions. More than 97% of the earth's water is salty; around 2% is frozen in glaciers and polar ice caps and less than 1% fresh water is within human reach [1]. Even this small fraction is believed to be adequate to support life and vegetation on earth.

Large areas of our planet suffer from lack of or pollution of fresh water. These include traditionally dry regions, such as deserts and modern industrial areas. The same problem also exists in remote areas and islands, where in many cases fresh water supply by means of transport is expensive. One study of water scarcity trend estimates that approximately 6.3 billion people live on earth, of which 400 million people now live in water scarce areas and this number could grow to 4 billion by mid-century.

Water pathogens, water turbidity, local population density, availability, existing water disinfection, community structure, infrastructure issues, sanitation practices, hygiene practices, income and awareness of diseases are some of the reasons for water pollution, which have decreased the total amount of fresh water available for human consumption [2].

The textile and tannery industries are facing lot of problems to dispose their effluents. Since these effluents

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are salty, their treatment is expensive. Usually they dispose it with help of natural evaporation ponds. The land area required for such ponds are very high. Getting land nearby such industries are also very difficult. The population density is too high in an industrial area. Many research works are being carried out to effectively dispose the industrial effluents with [3–8] and without [9] the help of solar energy. Potoglou et al. [10] distillated the olive mill waste water. Scrivani et al. [11] used solar energy for fresh water production and waste water treatment. In this work, it is planned to desalinate the industrial effluent water using solar desalination techniques to produce fresh water. The desalinated water may not have the required minerals. Many tests [5] are being conducted to check the quality of the distilled water. An under ground rock bed, green house heating was conducted by Kurklu et al. [12] to store solar energy. Salihoglu [13] carried out a study to investigate an economical solution to the sludge management problem. Santos et al. [14] proposed a design procedure for sizing solar-assisted crop-drying systems and assessing the combination of solar collector area and auxiliary energy needs that meet the requirements of the load. Yiannopoulos et al. [15] designed a solar-heated reactor system to enhance the anaerobic treatment of wastewater or biological sludge at temperatures higher than the ambient air temperature. In most of the previous research work, raw water or salty water is used as feed in solar desalination. In this work, effluent from textile industries is taken as input. Being an effluent a settling tank is used to get clarified liquid. This clarified liquid is sent to solar stills for desalination. The chemical properties are analysed for raw effluent, clarified effluent from settling tank and desalinated water from solar still. The cost of the minerals to be added in the distilled water is also estimated and based on this cost analysis were done.

2. Experimentation

The quantity of minerals present in the industrial raw effluent is too high. The raw effluent cannot be used in solar stills directly, as it is corrosive in nature. For settling and to reduce the quantity of unwanted minerals in the raw effluent, a settling tank is fabricated. Gupta and Chaudhuri [17] used sand, charcoal and gravel for purifying the salt water. In this work, the settling tank is fabricated with five layers namely tray for raw effluent, pebble layer, coal layer, sand layer and collection tray for settled effluent. The settled effluent is collected in a storage tank, which is placed beneath the settling tank. A PVC tank with 50 liters capacity is used as storage tank. From the storage tank, the settled effluent is sent to the solar still for desalination as shown in the Fig. 1.

The raw effluent settles in the settling tank and the settled effluent will be collected in the storage tank. From the storage tank, the clarified effluent is passed to a solar still through a polyvinyl chloride (PVC) hose pipe.

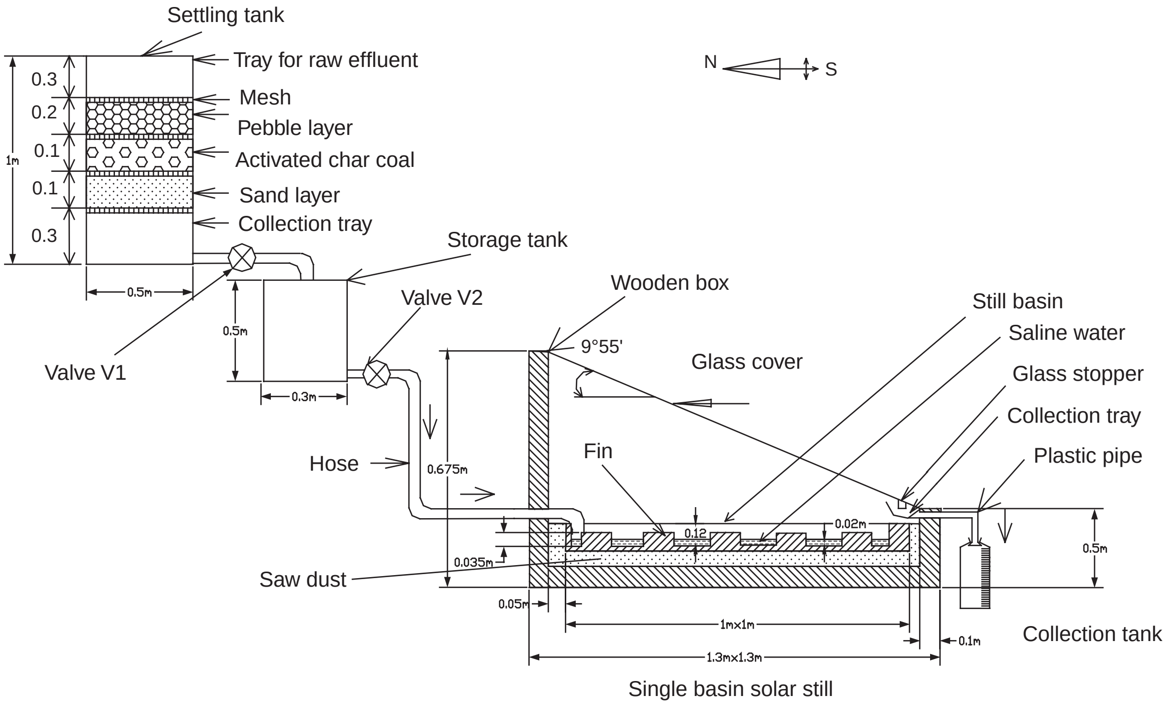

Fig. 1. Solar still integrated with an effluent settling tank.

A measurement system has been installed for recording the experimental data. The temperatures of the basin, water and glass cover in solar still are measured by means of Copper Constantine thermocouple connected to a digital meter through a selector switch. Solar radiation is measured with calibrated Kipp-Zonen Pyranometer. The amount of produced potable water is measured by a measuring beaker of 1 litre capacity. Wind velocity is measured by a digital vane type anemometer.

The productivity of the solar still is very less. To enhance the productivity of the solar still, many research works are being carried out [18–23]. It is found that, the maximum productivity occurred when a depth of 2 cm was maintained in the solar still. Hence, the saline water from the storage tank is filled in solar still up to a depth of 2 cm by opening the valve 'V'. The saline water gets heated and evaporates by solar energy. The evaporated water vapour is condensed at the inner side of the glass and glided along the glass. It is then collected in a beaker through a collection tray. The readings for distillate water yield, glass temperature, water temperature, basin temperature, wind velocity and solar intensity are taken for every one hour. During evaporation, the concentration of still water rises. To minimize the rise in concentration, the make up water is added for every half an hour. This experimental setup is designed, installed and tested at Thiagarajar College of Engineering, Madurai, Tamil Nadu, India.

3. Chemical analysis

For determining the various minerals present in the effluent, settled effluent and distilled water samples, the following measurement techniques were adopted.

3.1. Determination of electrical conductivity

The electrical conductivity of water samples are determined from the Elico conductivity bridge with null indicator type CM82T instrument provided with conductivity cell of cell constant 1. The instrument is calibrated with standard 0.1 Normality of potassium chloride (KCl) at 30C and the conductivity is measured by dipping the washed and rinsed conductivity cell into the water sample.

3.2. Determination of total dissolved solids

Total dissolved solid is determined from the residue left after evaporation and drying the water sample. 100 ml of the water sample is taken in a previously weighed beaker (W_1) and heated on a hot plate. After the residue is formed, the beaker is dried in an oven at 110C for an hour, cooled in a dessicator and weighed to a constant value (W_2). This process is repeated in duplicate, until a constant weight is obtained in each case. Total dissolved solids (TDS) of the water samples is arrived at from the weight of the residue using the equation:

$$\text{TDS in mg/l} = \frac{(W_2 - W_1)}{\text{volume of sample}}$$

Average value of the TDS for each water sample was found out and reported.

3.3. Determination of pH

pH of the water samples are measured with digital pH meter of Elico make model LI-120 using a coupled glass gel-filled electrode and a calomel reference electrode of pH range 0–14. Calibration of pH meter is done prior to measurement with a standard phosphate buffer of pH 7. The washed and rinsed electrode is dipped into the water sample taken in a beaker and pH value is noted down after the attainment of equilibrium.

3.4. Determination of total alkalinity

50 ml of the water sample, treated with 2 drops of methyl range indicator is titrated against standard H_2SO_4 until the colour changed from orange to yellow. The titration is repeated for concordant value and the volume of H_2SO_4 required (V_2) is noted down.

$$\text{Methyl orange alkalinity (mg/l as } CaCO_3) = \{\text{Volume of } H_2SO_4 \times \text{strength of } H_2SO_4 \times \text{equivalent weight of } CaCO_3\} / \text{Volume of sample}$$

3.5. Determination of acidity

100 ml of the sample is pipetted out into a conical flask. Three drops of phenolphthalein indicator is added. The solution is titrated against 0.02 Normality of sodium chloride (NaOH) solutions taken in a burette. The end point is the appearance of permanent pale pink colour.

3.6. Determination of total hardness

The method employed is complexometric titration of water sample with the disodium salt of Ethylene diamine tetraacetic acid (EDTA) using Eriochrome Black-T, the visual indicator. The samples are buffered with NH_4OH–NH_4Cl to a pH of 10. The EDTA is standardized using 0.01 Molar Ca^{2+} solution. In a typical procedure, 20 ml of the water sample is pipetted out into a clean conical flask. It is treated with about 2–3 ml of NH_4OH–NH_4Cl buffer solution. The solution mixture is titrated against standard

EDTA slowly with constant stirring using Eriochrome Black-T indicator. The end point of the titration is detected from the colour change of wine red to blue. The titration is repeated for concordant values and the volume of EDTA was noted. Total hardness is determined as,

Total hardness as $CaCO_3$ in mg/l = volume of EDTA × strength of EDTA × 1000/ volume of sample

3.7. Determination of calcium

Determination of calcium is made by following the standard procedure. 50 ml of sample, treated with one litre of 1Normality of Hydrochloric acid (HCl) and boiled for a minute, cooled and 2 ml of 1 N NaOH is added. Then it is titrated against, standard EDTA using Eriochrome black-R indicator. The end point is change of colour from red to blue. The titration is repeated to find a concordant value. The initial and final readings are noted as T_1 and T_2.

Amount of calcium in mg/l = $(T_2 - T_1) \times 20/(T_4 - T_3) \times (1/V)$

T_4 and T_3 are the readings obtained for control standard. Control standard is made by taking 20 ml of control standard (1ml of solution contains 1 mg of Ca) then 1ml buffer, 1 ml sodium sulphide inhibitor and 1 ml calmagite indicator are added then titrated against standard EDTA. The end point is the change of colour from pinkish red to blue. The initial and final titre readings noted as T_3 and T_4.

3.8. Determination of magnesium

Magnesium is determined by volumetric method. 20 ml of the sample is taken in a 250 ml clean conical flask and diluted it with 25 ml of double distilled water. 2 ml of ammonium chloride–ammonia buffer solution and 30–40 mg of Eriochrome Black-T indicator are added. Then the mixture is titrated with 0.02 M EDTA solution until the colour changes from wine red to steel blue. No tinge of reddish blue should remain at the equivalence point.

Amount of Mg (mg/l) = $0.00024 \times V/20$

where V = volume of 0.02 M EDTA used for magnesium.

3.9. Determination of Sodium

Sodium is determined by using flame photometer. At first, the meter is set to '0' reading with double distilled water. Then the meter is set to '100' reading with Na standard (Na = 10 mg/l). Then, the sample is injected into the meter and the readings are noted.

Amount of sodium in mg = reading from graph (for Na reading) mg/l × dilution factor.

3.10. Determination of potassium

The same procedure is repeated in this section and amount of potassium is calculated as,

Amount of potassium in mg = reading for potassium × slope × dilution factor

3.11. Determination of ammonia

Ammonia present in the water sample is determined by Nesslerization method. One ml of $ZnSO_4$ solution is added to 100 ml of the sample and mixed thoroughly. The pH of the sample is adjusted to 10.5 using 6 N NaOH and the mixture is allowed to stand for a few of the sample is taken in Nessler tube. 0.05 ml EDTA solution is added to the sample and mixed well. To this 2 ml Nessler reagent is added and allowed to stand for few minutes. The absorbence is measured at 420 nanometer using 1 cm cell with a reagent blank.

3.12. Determination of phosphate

The phosphate in water sample is boiled with ammonium molybdate which formed a complex heteropolyacid which is reduced to a complex blue colour in the presence of stannous chloride. The absorption of light by this blue colour is measured at 690 nm spectrophotometrically to calculate the concentration of phosphate.

Phosphate (mg/l) = OD for sample × slope × 50 / volume of sample taken for digestion

3.13. Determination of nitrite

Nitrite present in the sample can be measured using calorimeter. For 25 ml of the sample, 1 ml of colour reagent is added and stirred, then OD at 540 nm is measured after 10 minutes. Before measuring the OD for the sample, zero setting is done by 25 ml of double distilled water and 1 ml of colour reagent.

Amount of nitrite (mg/l) = OD1 × slope × (50/V)

3.14. Determination of nitrate

This is estimated by using UV-Vis-spectrophotometer–SL–159. All the samples are estimated using Brucine method. A range of concentration varying from 0.020 to 1.000 mg of N is set by preparing a series of 50 ml standard solution of the nitrate, from the stock standard nitrate. Two ml aliquots of standard solution are pipetted into different 100 ml beakers provided with glass rods to which 1.0 ml brucine sulfanilic acid and 10 ml sulphuric acid solution are added. It is placed in the dark for 10 min after stirring thoroughly when it develops yellow colour. Then it is stirred again after adding 10 ml of distilled water to each of the beakers and placed in dark for 30 minutes. The absorbance of each solution is then measured at 410 nm. A graph is plotted with absorbance value against the nitrate solution concentration of the standard solutions. 10 ml water samples are subjected to the same treatment as above and their absorbance is noted followed by the plotting of the graph with absorbance versus nitrate concentration. Care is taken to clean and dry the Nessler's tubes before insertion into the spectrophotometer.

3.15. Determination of sulphate

Determination of sulphate is made by following the standard procedure. The gravimetric method consists in adding a dilute solution of barium chloride to the water sample and thus converting all the sulphate into insoluble barium sulphate precipitate. In a typical procedure, 100 ml of water sample is pipetted out into a clean 500 ml beaker and acidified with few ml of 0.5 Molarities of HCl. The solution is slightly heated and to the solution about 15 ml of 0.1 Molarities of barium chloride is added from a burette drop wise with constant stirring. The precipitate formed is allowed to settle on steam bath for an hour. The supernatant liquid is tested for the presence of any free sulphate ion by adding few drops of barium chloride solution. The precipitate is filtered through a previously weighted sintered crucible, washed with warm water and dried in air oven at 120C to constant weight. Amount of sulphate in the water sample was determined from the weight of precipitate using the equation:

Sulphate (mg/l) = (weight of precipitate × molecular weight of sulphate)/molecular weight of barium sulphate × volume of water

The experiment is conducted in duplicate and the average value of sulphate is reported.

3.16. Determination of chloride

The presence of Chloride in water sample is determined volumetrically by the method called the "Mohr Method". 100 ml of water sample is pipetted into a clean conical flask. 1 ml of potassium chromate indicator is added and the water sample is titrated against standard $AgNO_3$ (0.014 N) till the colour changed from yellow to brick red. The titration is repeated for concordant values and the volume of silver nitrate is noted (A). The same titration is performed with deionised distilled water in the same way and again the volume of silver nitrate is noted (B). The amount of chloride in each water sample is determined from the equation,

Chloride (mg/l) = (A–B) × strength of silver nitrate × 35.46/volume of sample

3.17. Determination of fluoride

The fluoride content of water samples is measured by adopting colorimetric technique in a fluoride meter of Elico make mode CL 352. A calibration curve of absorbance versus concentration of fluoride using appropriate blank and standard sodium fluoride solution is generated in the instrument itself. Aliquots for fluoride measurement are prepared by adding 5 ml of the water sample and 1 ml of the calorimetric reagent supplied by the instrument manufacturer into 10 ml of volumetric flask and making the solution to the mark with deionised distilled water. These are shaken well, allowed for sometime to stand and introduced into the cell chamber. Fluoride concentration in ppm is displayed directly in the read out of the instrument.

3.18. Determination of salinity

The chlorinity of a sample of water has a definite relationship with salinity and hence the salinity can be determined from chlorinity using the Knudsen's equation.

Salinity (mg/l) = 0.03 + (1.805 × chlorinity of the sample)

3.19. Determination of dissolved oxygen

The determination is based on the Winkler method. In a typical procedure, 125 ml of water sample is taken in a 125 ml of BOD bottle and is treated with 0.8 g of $MnSO_4$ solid (corresponding to 2 ml of 36% $MnSO_4$ solution) followed by 2 ml of alkali iodide–azide reagent. The sample is shaken well without the entry of outside air into it and kept aside for sometime until all oxygen dissolves in the sample reacted with $MnSO_4$. After the formation of clear supernatant liquid, the contents are transferred to a conical flask. Immediately the BOD bottle is rinsed with 2 ml of concentrated H_2SO_4 which is also transferred to the same conical flask. The contents are then titrated with standard sodium thiosulphate iodometrically. This procedure is repeated to an almost concordance with the titre value.

The sodium thiosulphate had a strength of 0.0255 N and hence 1 ml of sodium thiosulphate is equivalent to 1.02 mg/l of dissolved oxygen.

3.20. Determination of biochemical oxygen demand

This determination is based on the measurement of dissolved oxygen (DO) of water sample before and after oxidation of organic matter of the sample by microorganism in an incubation period of 3 days at 27C. Winkler method is the chemical technique involved in the measurement of DO.

About 500 ml of the sample water is aerated at room temperature for 3 hours in a gas wash bottle. Nutritive reagents for bacterial growth are not added as they are not found to affect the final results significantly. Seeding of the sample is not made as the sample, being surface water contained sufficient bacterial population. Two aliquots of volume 125 ml each are withdrawn and their initial DO are determined as described previously and the average DO is noted down (D_o).

Two other aliquots of the aerated sample are also withdrawn and incubated in air tight plastic containers at 27C for 3 days in a cryostatic water bath. The bath DO of the incubated samples is determined as usual and the average final DO is noted down (D_3). A blank experiment with distilled dilution water in the place of water sample is conducted under identical conditions and its initial and final DO's are also measured (C_o and C_3 respectively).

BOD of the sample (mg/l) = $[(D_o - D_3) - (C_o - C_3)]$

3.21. Determination of chemical oxygen demand

This technique has the principle of chemical oxidation of organic matter of water sample by potassium dichromate in the presence of about 50% H_2SO_4 and determination of excess dichromate by titration against standard ferrous ammonium sulphate (FAS).

50 ml of sample water taken in a round bottomed flask is treated with 10 ml 0.25 N of potasium dichromide ($K_2Cr_2O_7$), 75 ml of concentrated analytical reagent H_2SO_4, 1 gm of Ag_2SO_4, and 1 gm of $HgSO_4$ and is shaken well. The contents are refluxed for 2 hours with a water condenser and cooled down. The contents are transferred and made up with distilled dilution water to 250 ml in a standard measuring flask. The excess dichromate is titrated against standard FAS using ferroin indicator until the colour of the solution changed from blue-green to wine red. The titration is repeated for concordant value (B). The blank experiment in the same manner is done with distilled dilution water and the volume of FAS required is also noted down (A). COD of water sample is calculated from the equation.

$$\text{COD (mg/l)} = (A-B) \times \text{strength of FAS} \times 8/ \text{ volume of sample}$$

In all the cases, the experiments are conducted twice and in many cases thrice so as to contain the error within the minimum level possible when there is considerable deviation in the measured values of any parameter, the average value is calculated and presented.

4. Addition of minerals

It is found that the minerals present in the desalinated water are very low. The acceptable range of minerals and available minerals in distilled water are given in Table 1. To make the water potable, some minerals are added as per the guidelines given by the TWAD board. The shortage of calcium in the distilled water is 55–180 mg/l. While adding 400 mg of $CaCl_2$, 144 mg of Ca and 254 mg of Cl_2 will increase in the saline water [3,7]. Similarly the other required minerals are to be determined and added as per Table 2.

Table 1
Shortage of minerals in desalinated water.

Sl. No.	Parameter	Acceptable range of limits as per TWAD guidelines (mg/l)	Available minerals in distilled water at stills (mg/l)
1	Total alkalinity	200–600	66
2	Total hardness	200–600	60
3	Calcium	75–200	20
4	Magnesium	30-150	2
5	Iron	0.1–1	0.07
6	Manganese	0.05–0.5	0.00
7	Nitrate	45–100	0.00
8	Chloride	200–1000	8
9	Fluoride	1–1.5	0.00
10	Sulphate	200–400	3

5. Results and discussion

The properties of raw effluent, settled effluent and distilled water are calculated by physical (turbidity, total dissolved solids) and chemical analysis (pH, alkalinity, total hardness, calcium, magnesium, potassium, sodium, iron, manganese, ammonia, nitrate, chloride, fluride, sulphate, phosphate, BOD and COD) at Tamil Nadu Water supply and Drainage board (TWAD), a government sector, Tamil Nadu, India. Upper and lower limits of various minerals, that can be present in potable water is also given by TWAD board. Based on this, some minerals are added to convert the produced distilled water into mineral water. The various minerals present in raw effluent, settled effluent and distilled water are shown in the Fig. 2. It is found that around 99% of minerals are removed during settling and distillation. The cost of minerals to be added is given in Table 2.

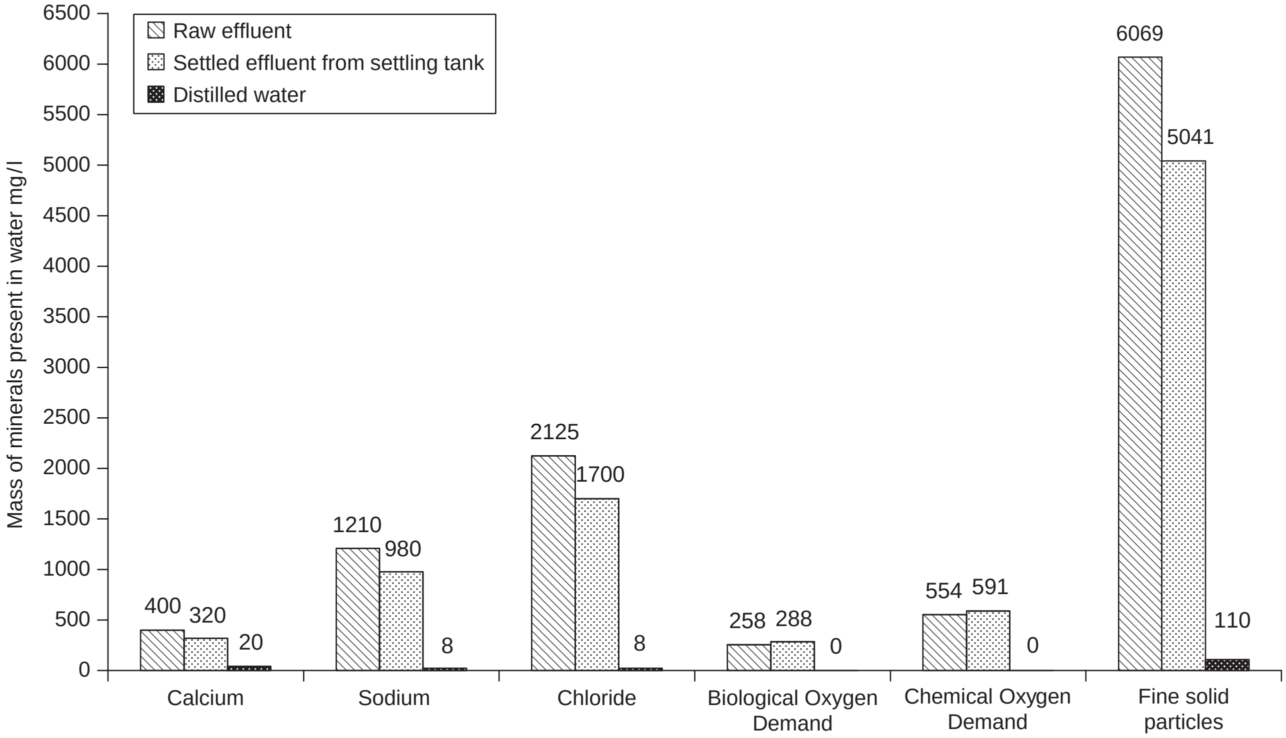

Fig. 2. Chemical analysis of raw effluent, settled effluent and distilled water.

Table 2

Mass of minerals added and their costs.

Sl.No.	Chemicals added		Increase of minerals (mg/l)										cost, paise
	Name	mass, mg/liter of water	Ca	Cl_2	$CaCo_3$ Total alkalinity	$CaCo_3$ Total hardness	Mg	So_4	Fe	No_3	Mn	F_2	
1	$Cacl_2$	400	144	255	-	-	-	-	-	-	-	-	6.4
2	$Ca(OH)_2$ $CaCo_3$	148 200	-	-	200	-	-	-	-	-	-	-	3.5 3.2
3	$CaCo_3$	400				400							6.5
4	$MgSo_4$	500	-	-	-	-	100	400					8.5
5	$Fe(NO_3)_3$	4.33							1	3.33			0.1
6	$MnSO_4$	1.37	-	-	-	-	-	0.87	-	-	0.5	-	0.1
7	CaF_2	2.05	1.1	-	-	-	-	-	-	-	-	1	0.1
	Total	1255.7	145.1	255	200	400	100	400.87	1	3.33	0.5	1	28.4

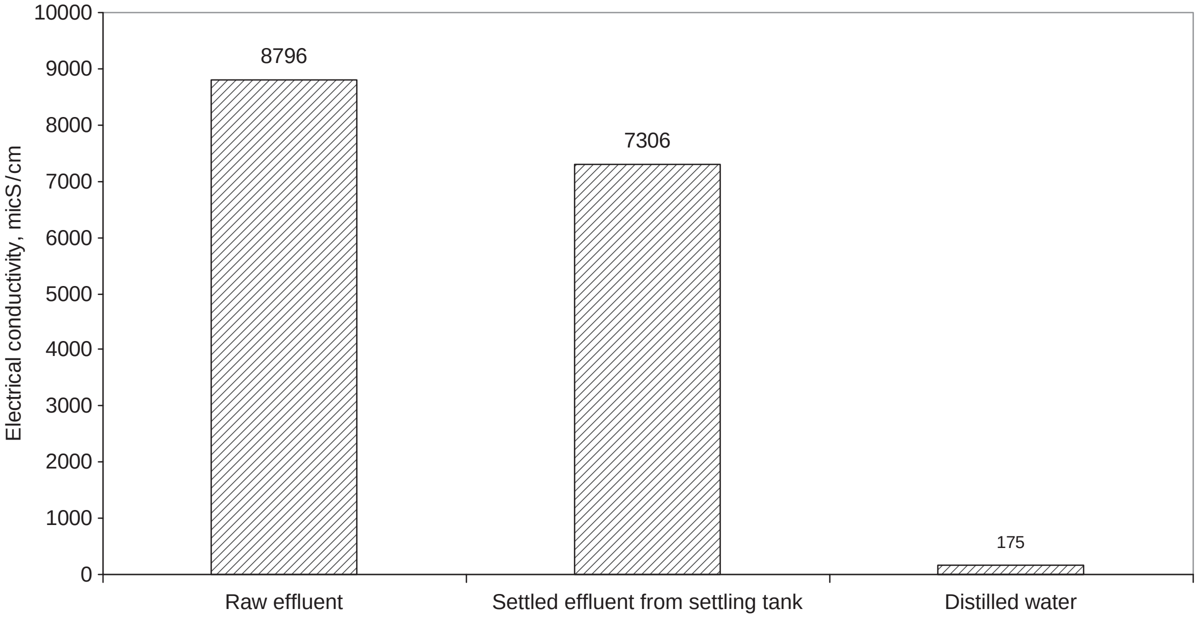

Fig. 3. Variation of electrical conductivity for various samples.

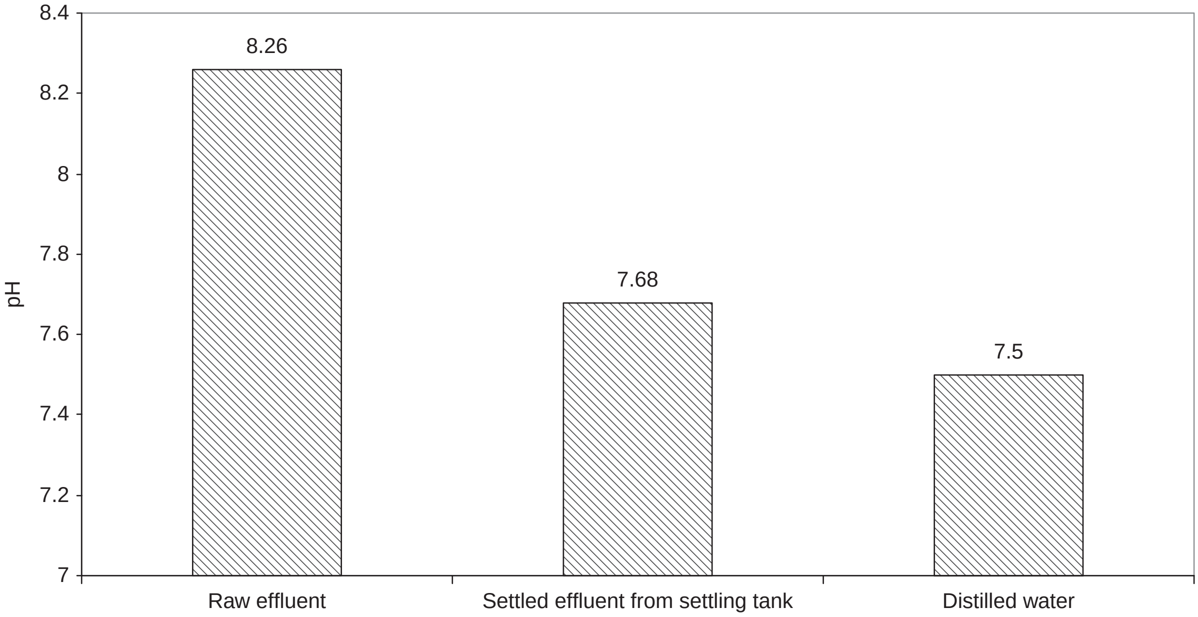

Fig. 4. Variation of pH for various samples.

Table 3
Error analysis.

Sl. No.	Instrument	Range	% Error
1	Pipette	0–20 ml	±1%
2	Burette	0–50 ml	±1%
3	pH meter	0–14	±1%
4	Conductivity meter	20 µS/cm–200 mS/cm	±1%
5	CM82T instrument	20 µS–200 µS	±0.01 µS
6	UV-Vis-spectrophotometer SL-159	192–1100 nano meter	±0.1 nano meter
7	Fluoride meter of Elico make mode CL 352	0.05 mg/lit–1.9 g/lit	±0.02 mg/lit

The electrical conductivity for the various samples is plotted in the Fig. 3. It was found that the electrical conductivity decreases when the effluent water is settled. The electrical conductivity decreases again when it is desalinated. The same effect is found for pH as shown in the Fig. 4.

6. Error analysis

Error analysis is done for various measuring instruments—pipette, burette, pH meter and conductivity meter are used for the chemical analysis. The error analyses for the instruments are shown in the Table 3.

7. Cost analysis

The cost analyses for other modifications are given in Table 2. As per the guidelines given by the TWAD board, the above mentioned minerals are added with desalinated water. The cost of the minerals is given in Table 2. The cost of minerals to be added per liter of water is found as Rs. 0.28 ($0.0056).

The payback period of the experimental setup depends on the overall cost of fabrication, maintenance cost, operating cost and cost of feed water. The cost of feed water is negligible. Fabrication costs include cost of glass, saw dust, galvanized iron sheet for making basin, wooden box, tank, hoses, collection tank, stand and labor cost.

The overall fabrication cost = Rs. 8000 ($160)

Cost per liter of distilled water = Rs. 10 ($0.2)

Mean daily productivity of the solar still (for a mean solar insulation of 700W/m^2) = $2.77\ \text{l/m}^2$

Cost of water produced per day = Rs. 27.7 ($0.554)

Maintenance cost = Rs. 5/day ($0.1)

Cost of minerals per liter = Rs. 0.2 ($0.004)

For 2.77 l = Rs. 0.55 ($0.011)

Net earning = cost of water produced – maintenance cost – cost of minerals Rs. (27.7 – 5 – 0.55)
= Rs. 22.15 ($0.443)

Payback period = Investment/net earning
= 8000/22.15 = 361 days

8. Conclusion

To solve the problem of disposing industrial effluent and water scarcity in industrial areas, an attempt is made to desalinate the effluent by solar desalination methods. The water samples from raw effluent, settled effluent and desalinated water are taken and tested. It was found that 99% minerals are removed during settling and desalting processes. As per the guidelines given by the TWAD board, some minerals are added to make the water potable. Cost analysis is done. The cost of minerals added per liter of water is 28.4 paise ($0.0056).

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