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COMPARISION OF PHYSICAL AND CHEMICAL PARAMETERS OF RAW SPENT WASH DISTILLERY WATER BEFOREAND AFTER NANO FILTRATION, WITH THAT OF REVERSE OSMOSIS HUMAN DRINKING WATER

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ABSTRACT

India is a vast country with many rivers and Tributaries. The Indian people for daily routine living life style habit rely largely on extreme use of water. Water is essential to every Indian house hold needs. Different methods are used to purify water, the methods applied were, Microfiltration (MF) method, Ultrafiltration (UF) method, Nanofiltration (NF) method and Reverse Osmosis (RO) method. In this article we have applied nanomembrane filtration method to purify sugar cane industrial effluents namely Raw Spent wash distillery effluent water diluted with Reverse Osmosis water. The physicochemical parameters were measured before and after Nanomembrane filtration in Raw spent wash distillery, as well as in Reverse osmosis filtered tapwater utilized by people for drinking purpose. There is a 40-70% of Rejection in chemical parameters in Raw spent wash distillery water after Nanomembrane filtration and physical parameter showed greater improvement. But the Rejection in chemical parameter by Reverse osmosis in Ordinary Tap water was found to be more than 85%. The physical parameters showed higher improvement in rejection than Nano filtered Raw spent wash water.

KEY WORDS : Reverse osmosis, Nanofiltration, Ultrafiltration, Micro filtration, Raw spent wash distillery

INTRODUCTION

There are several types of filtration methods applied. They are, a) Microfiltration Method b) Ultrafiltration Methodc) Nanofiltration Methodd) Reverse Osmosis Method, etc.

Microfiltration

Microfiltration is a process to purify water. Microfiltration method is used to separate *low molecular weight* suspended or Colloidal compounds from dissolved solids. Microfiltration separates compounds in the range of approximately 0.1 to 1 microns. Suspended particles are removed through microfiltration method.

Ultrafiltration

Ultrafiltration is exploited to separate *high molecular weight* components like plant and dairy proteins, carbohydrate polymers and enzymes. Ultrafiltration removes particles between 20 to 1000 Angstrom units upto 0.1micron. UF membranes have molecular weight ranges from 1,000 to 100,000. UF operates at a membrane pressure between 15 to 10 psi (1 to 7 bar).

Nanofiltration

Nano means 10^{-9}. Nanofiltration separate particles in the range of 1 nanometre (10 angstroms). In Nanofiltration molecular weight compounds between 200-400 molecular weights are rejected.

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Dissolved salts are filtered upto 20-98%. Sodium chloride (NaCl), Calcium Chloride ($CaCl_2$) are usually rejected upto 98%.

Nanofiltration is utilised to remove Total Organic carbon, hardness, radium from well water. The Nanofiltration utilises pressure between 50-225psi (3.5 to 16 bar).

Reverse Osmosis

RO membrane separates organic and inorganic molecules with a molecular weight greater than 100. Reverse Osmosis utilises 75psig for brackish water (5 bar) to greater than 1,200psig (84 bar) for sea water (Anil Kumar, *et al.,* 2014), (Baker, 2004)

Sugarcane industries were divided into the following categories.

1. Those Produces only raw table sugar
2. Those Produce only ethanol.
3. Those Produces both raw sugar and ethanol.

Globally 80% sugar industries are interested in producing both raw sugar and ethanol. The by-product of sugarcane industry is bagasse and Bioethanol. The waste product formed is stillage or vinasse or dunde, itis liquid in nature (Jemal *et al*., 2019).

The pore size, pressure, for the different membrane operation is given below.

The by-product obtained from Sugarcane industry is bagasse. Bagasse is the most significant by-product. After Sugarcane juice extraction the solid residue obtained is called bagasse the remaining part is called stillage. The stillage is also called Vinasse or dundes.

Bioethanol is the most valuable product that is obtained from fermentation and distillation of Sugarcane juice and molasses (Nandy, *et al.,* 2002).

In sugar industry during the preparation of sugar, molasses is formed along with molasses distillery waste water. The molasses distillery waste water is further divided into a) Fermenter Sludge b) Spent washc) Spentlees (Nataraj, *et al.,* 2006; Kharayat, 2012; Tewari *et al.,* 2007)

Fermenter Sludge

During ethanol separation or filtration fermenter sludge is generated, fermenter sludge is obtained due to fermentation of molasses.

Spent Lees

Spent Lees is a hot and colourless liquid obtained from rectifier column from rectified spirit in volume range from 1.7 – 2.0 l of rectified spirit.

Distillery spent wash

One of the higher amounts of waste water produced in ethanol distillery in sugar cane industry is named as spent wash. It is produced with certain amount of quantity with quality. It is also known as vinasse, stillage slop or molasses.

Spent wash that is produced is otherwise called as waste water, that is produced during the distillation process is always subjected to treatment.

The distillery spent wash effluent produced from molasses treatment have the following nature

1. Its COD is found to be 80,000-1,60,000 mg/L with high Organic matter.
2. It is naturally with high Temperature
3. It has high ash content
4. It has ow acidity (pH 3.7 - 4.5)
5. The inorganic salts content is very high
6. It has Deep brown colour

(Pant and Adholeya, 2007)

MATERIALS AND METHODS

The research study was carried out from sugarcane

Pore Size and Pressure for Different Filtration Methods

Membrane type	Pore size (nm)	Pressure (BAR)	Product water
Reverse Osmosis	0.6	30-70	Pure water (PW)
Nanofiltration	0.6-5	10-40	(PW)and low molecular solutes
Ultra-filtration	5-50	0.5-10	All above and macromolecules
Micro Filtration	50-500	0.5-2	All above and colloids

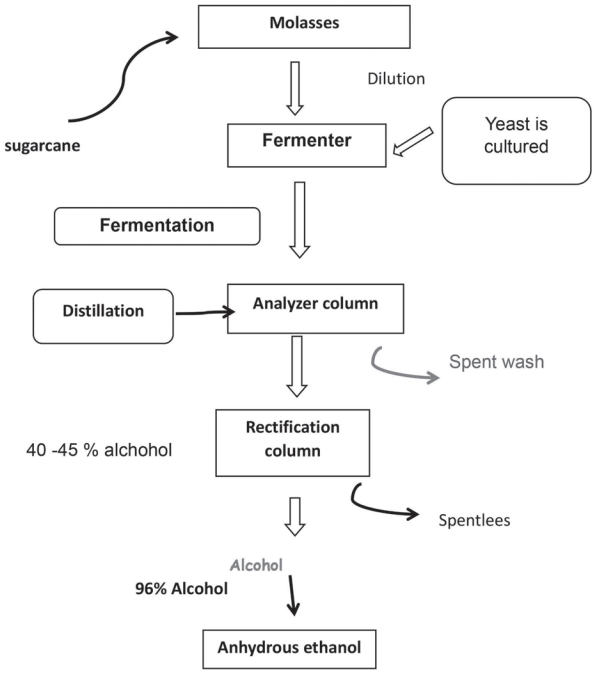


Fig. 1. Production of alcohol and spent wash in Sugarcane industry

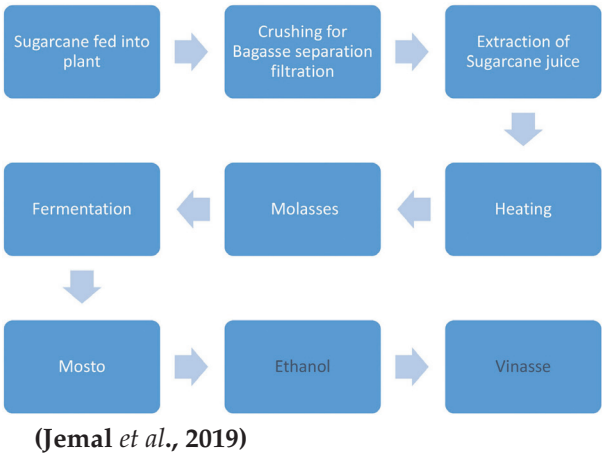


Fig. 2. Steps in the Production of Ethanol in Sugar Cane Industry

industries with different sugarcane effluents collected from *Sakthi Sugar Limited, SakthiNagar, Aapakudal, Erode District, 638315, India*

The effluents were collected in an airtight bottled sterilized from bacteria and taken for analysis within 4-6 hours.

The following effluents were collected from the refinery

1. Raw Spent wash water
2. Yeast Sludge water
3. Sugar waste water
4. Concentrate effluent distillery

We focussed our research on Raw spent wash water

The raw spent wash water was tested for the following physical and chemical parameters.

Physical parameters

Physical Parameters: Total solids, Total dissolved solids, Turbidity, Alkalinity Electrical conductivity
Chemical Parameters: Magnesium, Calcium, Phosphorous, Sodium and Sulphate Ultra filtration Protocol

Ultrafiltration operating pressure— 4-90 psi
Operating temperature—1-40 Degree Centigrade
pH of the membrane ——6-9

Nanomembrane Filtration Experiment Protocol

1. Nanomembrane operating pressure – 600psi
2. Nanomembrane operating temperature- 113°F
3. pH of the membrane -6-11
4. Allowed permeate flow – 225 GPD

Collection of water samples: The water samples were collected in a sterilised air tight container and taken for the analysis within 4 to 6 hours

The standard method adopted for testing of water were taken from APHA Manual which contains standard method for examination of water and waste water. All the standard testing for water is done at AWE care analytical and Research laboratories Erode-638012, Tamil Nadu India

EXPERIMENTAL SET UP

Dilution Procedure

12 l of Raw Spent wash water is taken and diluted to 8 l with ordinary Reverse Osmosis water (RO) and were subjected to different stages of treatment.

RESULTS

Table 1 shows the observation of results of Chemical parameters of Magnesium, Calcium, Phosphorous, Sodium and Sulphate in (mg/ml) in sugar cane raw spent wash distillery before Nano filtration and after Nanofiltration and Ordinary tap water Before RO and after RO

The decrease in both Nonfiltered Raw spent wash and Reverse Osmosis Tap water was found to be in the following order as given below in the table

Magnesium < Phosphorous < Calcium < Sulphate < Sodium

In stage I the Raw Spent wash water is collected and subjected to high-speed centrifugation in order to obtain a clear solution.

Stage-II

In stage-II process the Raw Spent wash water is subjected to double distillation and the collected water is cooled.

Stage-III

In stage-III process the water is diluted with 2Litre double distilled water and subjected to Ultrafiltration

Stage-IV

The finely ultrafiltered water is subjected to nanofiltration through nanofiltration membrane (**For every run 12 Litre of Sugarcane Raw spent wash water+ 8 litre water RO water is taken for the experiment in the beginning).**

Fig. 3. Experimental Design

THE FINAL RESULT OF CHEMICAL PARAMETERS OF RAW SPENT WASH AFTER NANOFILTRATION AND ORDINARY TAP WATER AFTER REVERSE OSMOSIS* (Table 3)

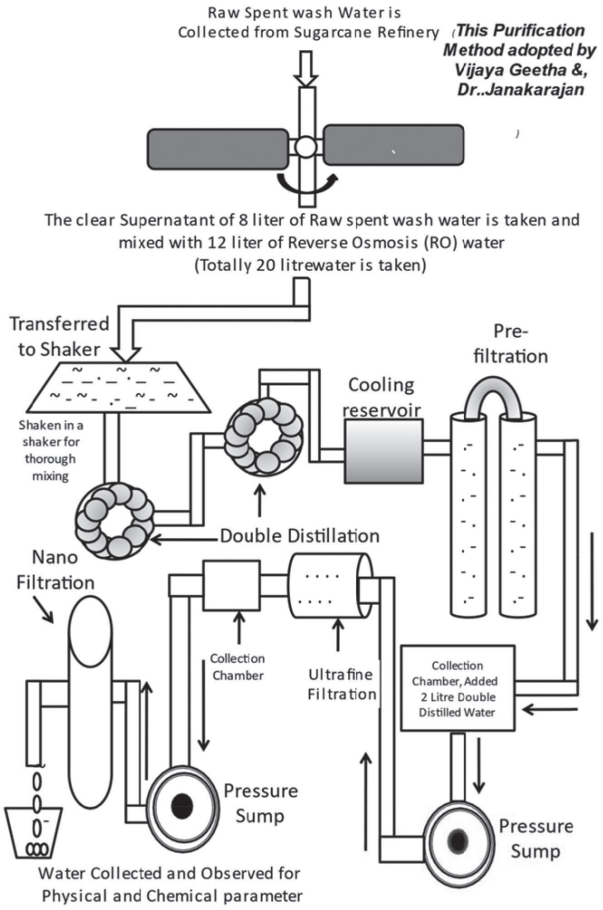
S. No.	Parameters	Raw spent wash After Nano Filtration (mg/L)	Ordinary Tapwater After Reverse Osmosis (mg/L)
1.	Magnesium	28	4.8
2.	Phosphorous	70	30
3.	Calcium	80	28
4.	Sulphate	60	30
5.	Sodium	1428	20

*The above Final observation is recorded as such

The decrease was found to be maximum in Reverse Osmosis filtered Tap water when compared to Raw spent wash Nano filtered water

Graph 1 & 2 represent the level of Sugar cane Raw spent wash distillery before Nanofiltration and After Nanofiltration. Graph 2 represents before RO and after RO in chemical parameters

Table 2 Represents the level of Chloride before Nanofiltration and after Nanofiltration in Raw spent



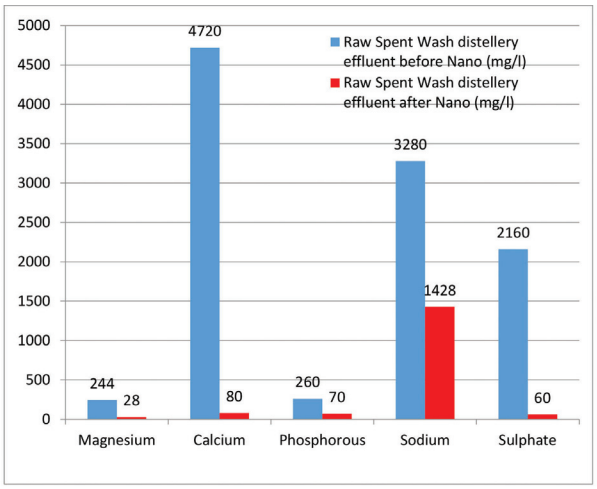
distillery spent wash and Reverse Osmosis water. It was found that the rejection of chloride was found to be maximum in Reverse osmosis water

Table 3 shows the observations on physical parameters in raw spent wash water before and after Nanofiltration and ordinary Tap water Before and after Reverse Osmosis

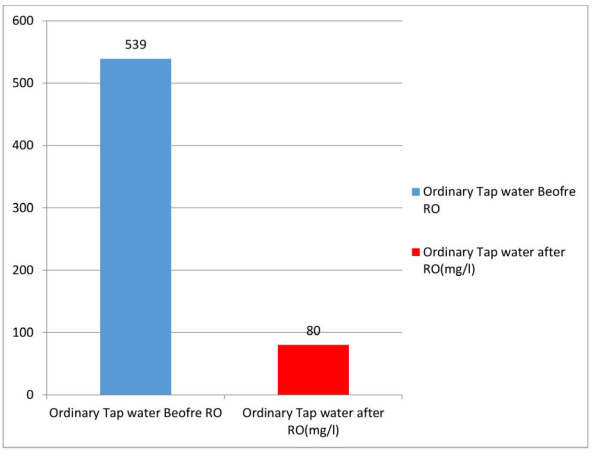
From the table it can be observed that the level of Turbidity, Total alkalinity, Total dissolved solids (TDS), and Total solids was found to be decreased to

Table 1. Our observation on Chemical parameter of Raw spent distillery spent wash effluent Before Nano filtration and after Nanofiltration and Ordinary tap water before Reverse Osmosis (RO) and After Reverse Osmosis (RO)

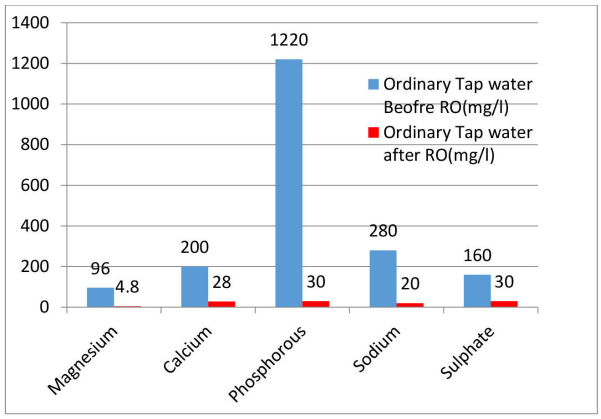
S. No.	Parameters	Raw Spent Wash distillery effluent before Nano Filtration (mg/L)	Raw Spent Wash distillery effluent after Nano Filtration (mg/L)	Ordinary Tap water Before RO (mg/L)	Ordinary Tap water after RO (mg/L)
1	Magnesium	244	28	96	4.8
2	Calcium	4720	80	200	28
3	Phosphorous	260	70	1220	30
4	Sodium	3280	1428	280	20
5	Sulphate	2160	60	160	30



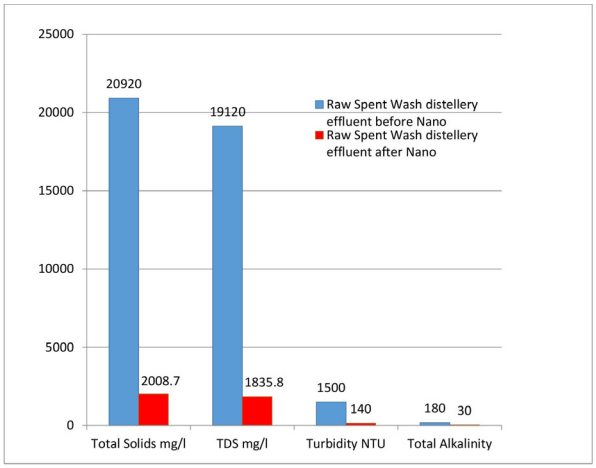
Graph 1. Comparison of Chemical parameters of Magnesium, Calcium, Phosphorous,Sodium, Sulphate of Raw spent wash Distillery Effluent before Nano Filtration and After Nano Filtration



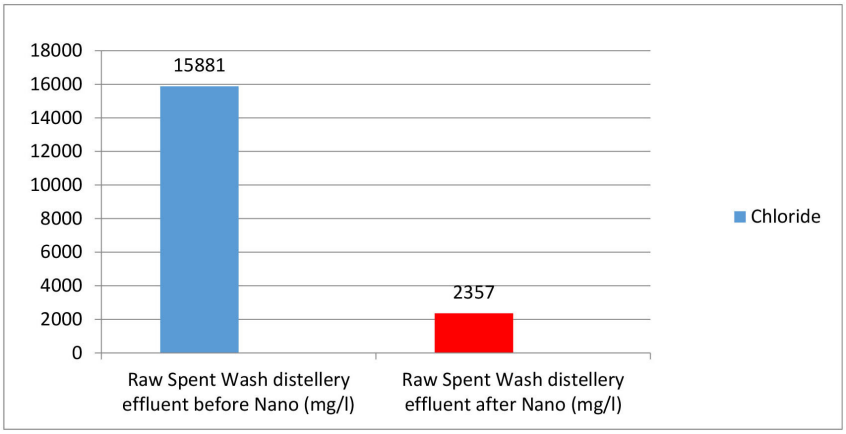
Graph 4. Comparison of Chemical parameter, Chloride level (mg/l) on Tap water Before Reverse Osmosis and After Reverse Osmosis



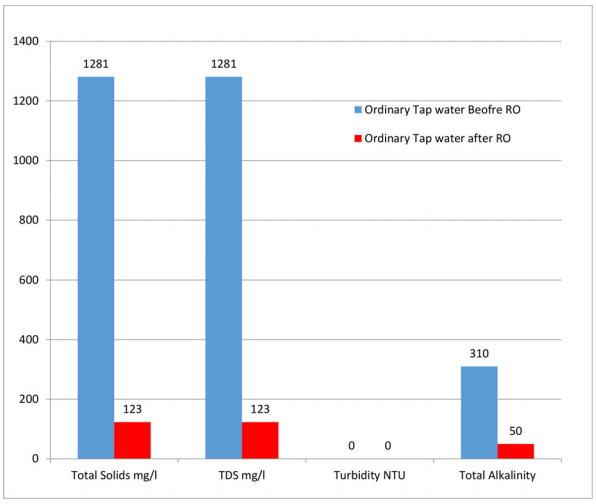
Graph 2. Comparison of Chemical parameters of *Ordinary tap water* Before Reverse Osmosis and After Reverse Osmosis on Magnesium, Calcium, Phosphorous, Sodium and Sulphate



Graph 5. Observation on Physical parameters on Raw spent wash distillery before Nano filtration and after Nano filtration



Graph 3. Comparison of Chloride level before Nanofiltration and After Nanofiltration in Raw spent wash distillery effluent



Graph 6. Observation on Physical parameters on Tap water before Reverse Osmosis and after Reverse Osmosis

a maximum in Reverse Osmosis Tap water when compared to Raw spent wash distillery Effluent after nanofiltration.

The decrease in the physical parameters was found to be in the following order as given in the Table in both Nano filtered Raw spent wash distillery Effluent water after Nanofiltration and Tap water After Reverse Osmosis

Turbidity< Total alkalinity< Total dissolved solids d" Total solids

THE FINAL RESULT OF PHYSICAL PARAMETERS OF RAW SPENT WASH AFTER NANOFILTRATION AND ORDINARY TAP WATER AFTER REVERSE OSMOSIS*(Table 3)

Table 2.

S. No	Parameters	Raw spent wash Distillery Effluent After Nan ofiltration	Ordinary Tap water After Reverse Osmosis (RO)
1.	Total solids mg/l	2008.7	123
2.	TDS mg/l	1835.8	123
3.	Turbidity NTU	140	0
4.	Total Alkalinity (mg/l)	30	50
	Total hardness	240	90

*The above final observation is recorded as such

***E coli* test gave negative results in Raw spent distillery wash before and after Nano filtration

Graph 5 & 6 shows the level of Total solids, Total dissolved solids (TDS), Turbidity, and Total alkalinity in Raw spent wash distilled water before and after Nanofiltration

DISCUSSION

Nanomembrane is mainly governed by the following factors a) Pressure b) temperature c) Biofoulingd) feed characteristics and permeate flow.

The importance of Bio membrane fouling is discussed by (Shon *et al.*, 2005). He stressed the importance of pre-treatment. The fouling occurs on the surface of both Nanofiltration membrane and in Ultrafiltration membranes. The organics in the foulant and in membrane increased before pre-treatment, afterpre-treatment it was found to be lowered.

Nanomembrane seperates particles in the range size between 0.001—0.1-micronsize. The Raw spent wash that has been procured from sugar cane industry, contains higher turbidity, total solids, total dissolved solids. The high turbidity and total solids may damage the nano membrane structure

In order to increase the nanomembrane efficiency and permeate flow, before nanomembrane filtration, we have diluted the sugar cane raw spent wash water with Reverse Osmosis water (*12 Litres of sugar cane raw spent wash diluted to 8L Litre of double distilled water.)*

Table 3. Observation on Physical parameters on Raw spent wash distillery before Nano filtration and after Nanofiltration Ordinary Tap water Before Reverse Osmosis (RO) and After Reverse Osmosis (RO)*

S. No.	Parameters	Raw Spent Wash distillery effluent before Nano Filtration	Raw Spent Wash distillery effluent after Nano Filtration	Ordinary Tap water Before RO	Ordinary Tap water after RO
1	Total Solids mg/l	20920	2008.7	1281	123
2	TDS mg/l	19120	1835.8	1281	123
3	Turbidity NTU	1500	140	0	0
4	Total Alkalinity (mg/l)	180	30	310	50
5	Total hardness mg/L	22,000	240	900	90

*The final results were recorded as such

It is then boiled, cooled and prefiltered. Prefiltration and double distillation is done to avoid nanomembrane bio fouling.

In the final stage of purification process, we have also added *two litres of cooled double distilled water before Ultra filtration*

The pore size of Ultra filtration filter is around 0.01 micron.

Ultrafiltration removes particles in the range size of 20 to 1000 angstrom units (upto0.1 microns).An ultra-filtration filter has a pore size around 0.01micron. (US Environmental protection agency, 1999), (US Environmental protection agency, 2007), (WHO, 2004, Water Treatment and pathogencontrol: Process Efficiency in Achieving safe drinking water).

Distillery spent wash Characteristic

The distillery spent wash is a problematic product, its composition is discussed by (Muhammad Umair Hassan *et al*., (2021). It was found that Nitrogen, Phosphorous, Potassium, Calcium, Magnesium, Sodium, were found to be high. The chemical nature of the Raw spent wash distillery is found to be in the following order

Potassium > Sulphate >Calcium > Magnesium >Nitrogen > Sodium >phosphorous 17,475 mg/l, 3425 mg/l, 7000 mg/l, 2100 mg/l, 4200 mg/l, 670 mg/l, 3038 mg/l

Sulphate, Zinc, copper Iron, and Lead were found to be higher in Raw spent wash. The physical parameters of Raw spent wash like Total solids (TS) mg/l Total suspended solids (TSS) mg/l, and Total dissolved solids (TDS) mg/l was found to be in the following order (Rathi *et al.,* 2010).

Total dissolved soils >Total solids >Total suspended solid > Total settleable solids

27240 mg/l 35,340 mg/l 9980 mg/l 9860 mg/l

BOD-15,300 mg/L, COD-30,520 mg/l, Electrical conductivity (µs) -28,700 pH-7.23

The appearance of the Raw spent wash distillery was found to be brown in colour, the total hardness was found to be above 22,000 mg/l the turbidity was found to be above 1000 mg/l. The brown colour in Raw spent wash is caused by Melanoidins which causes dark brown colour. The molecular weight of Melanoidins is about 5000-40,000 Daltons and accounts to 2% total DWS (Distillery water) (Wang *et al.,* 2011; Yadav *et al.,* 2011; Prasad *et al.,* 2009)

Factors Governing Nanomembrane filtration

In Nano filtration the permeability and rejection are fully governed by the following factors a) Nature of thesolution b)Viscosityc) Solute diffusivityd) Solution pHe) Solution temperaturef) Solvent thicknessg) Membrane thickness h) Membrane dielectric constant.I) Membrane temperature. Nature of the solution that is to be filtered is more important. The pore size of the membrane is also important. The pressure and pore size of the membrane plays a crucial role in purifying water.

In our experimental set up we have also subjected the raw spent wash water to undergo Ultrafine filtration after adding 2 litres of water. This is done to avoid membrane damage

Our final observation on physico chemical parameters of Nano filtered Distillery Raw spent wash Effluent with that of Ordinary Tap water after Reverse Osmosis (Extracted from Results Table 1 and 3)

Table 4.

S. No.	Parameters	Raw Spent wash distillery Effluent After Nanofiltration	Ordinary Tap water After Reverse Osmosis
1.	Total solids (TSS)	2008.7	123
2.	Total dissolved solids (TDS)	1835.8	123
3.	Turbidity NTU	140	0
4.	Total Alkalinity	30	50
5.	Electrical conductivity	20	—
6.	Magnesium (mg/l)	28	4.8
7.	Phosphorous (mg/l)	70	30
8.	Calcium (mg/l)	80	28
9.	Sulphate (mg/l)	60	30
10.	Sodium (mg/l)	1428	20

**E coli test gave negative results in Raw spent distillery wash before and after Nano filtration

Absence of microorganism bacteria: The microorganisms Bacteria were completely absent before and after Nanofiltration. This might be due to double distillation and Nanofiltration.

(Rishikesh Singh *et al*., (2020) have studied about the removal of pathogens by using nano membrane technology in his study he could be able to completely observe that pathogenic microbes including bacteria, viruses and protozoa was completely eliminated due to nanomembrane filtration.

We have observed in Raw spent wash distillery after nanofiltration the physical parameters namely Total solids, Total dissolved solids (TDS), Turbidity, Total alkalinity, and electrical conductivity was

found to be decreased to a greater extent after Nanofiltration,

The chemical parameters Magnesium, Phosphorous, Calcium, Sulphate, and Sodium was found to be decreased to a greater extent after Nanofiltration in Raw spent wash distillery.

We would like to state that the decrease in physicochemical parameters in Nano filtered raw spent wash water is not to a greater extent as compared to Reverse Osmosis water.

OUR OBSERVATIONOF RAW SPENT WASH EFFLUENT DISTILLERY, AFTER NANOFILTRATION AND CONCLUSION

We have come across the following observation after Nanofiltration

1) The viscous force of the Raw spent distillery wash was found to be reduced
2) The electrical conductivity was reduced
3) The total turbidity
4) The total dissolved solids were reduced to a greater extent
5) The results were negative for the growth of bacterial colonies
6) The total solidswere found to be reduced
7) The Optical density of the raw spent wash was completely reduced the nanofiltration membrane performance was studied with several Electrolyte solutions with monovalent and divalent hardness ions like Potassium chloride, Calcium chloride and Magnesium Sulphate to find out the influence of these salts on pH andcharge on the membrane surface and also in membrane pores.

It was found that (a) There was an increase in pH with increasing in membrane negative charge (b) it was found that the membrane surface was more negatively charged than the pores (c) Membrane flux decreased with pH, whereas the retention capacity of the Nanomembrane increased with pH (d) When there is higher salt concentration (high ionic strength) flux and retention decrease

In our experimental set up the decrease in retention capacity of the Nanomembrane on Magnesium, Calcium, Chloride and Sulphate in Raw spent wash distillery might be due to higher salt content and ionic strength as observed by Margarida *et al.,* (2005) Umesh Kumarrai *et al.,* (2008) have observed the aerobically treated distillery waste water carried out in a spiral wound nanofiltrationmodule under the various operating conditions. It was found that a) The percentage separation of organic and inorganic compound is quite high b) Concomitant reduction in chemical oxygen demand was noted C) Total dissolved solids reduced to 96-99% and 85-95% respectively. D) The permeate flux was reduced, when the concentration of Feed solution increases

We could be able to achieve greater rejection of Magnesium, Sulphate, Phosphorous, Calcium and Sodium in Raw spent wash distillery. The rejection of Magnesium, Sulphate and Calcium was recorded to a greater extent when compared with initial concentration in Raw spent wash. The rejection of chemical parameters is not to a greater extent in Raw spent wash distillery wash when compared to Reverse osmosis water

The Nanofiltration lies in between Ultra filtration and Reverse osmosis, we could not able to achieve complete rejection of, Magnesium, Sulphate, Phosphorous, Calcium, and Total solids, total dissolved solids, Turbidity and alkalinity Even though we have diluted and distilled the Raw spent wash Distillery.

We have achieved more than 40- 70% rejection in physical and chemical parameters, but complete rejection could not be achieved, this might be due to the complexity of Raw spent wash Distillery that we have discussed earlier as it contains high turbidity and high dissolved organic matter. Still the complexity of Raw spent wash is not yet completely understood

Nano filtration cannot be done directly for sugarcane raw spent wash distillery water. The raw spent wash contains many impurities, and dissolved solids. It exhibits higher turbidity. Higher turbidity may damage the Nanomembrane.

The separation of particles in Nanomembrane filtration depends on (a) Pressure (b) Nature of the substance to be separated (c) concentration of the substance (d) Turbidity permeate flow

We have also observed the Raw spent wash contains higher amount of Total dissolved solids (TDS) and total solids (TS).

So, we have diluted the Raw spent wash with RO water and double distilled and prefiltered and also with pure double distilled water before Ultra filtration.

The result we have obtained after Nanofiltration is improved in physicochemical parameters (40-70%) but not to the complete extent as in Reverse osmosis drinking water

Advantage of our method: It is cheap and

Economical and less space consuming
Disadvantage of our method:- Double distillation takes longer time

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