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Recycling Monoethylene Glycol (MEG) from the Recirculating Waste of an Ethylene Oxide Unit

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Abstract: In the ethylene glycol generation unit of petrochemical plants, first a reaction of ethylene oxide takes place which is then followed by other side reactions. These reactions include water absorption with ethylene oxide, which leads to the generation of formaldehyde and acetaldehyde. Over the lifetime of the alpha-alumina-based silver catalyst there is an increase in side reactions, increasing the amount of the formaldehyde and acetaldehyde generated by the ethylene oxide reactor which leads to reduced MEG product purity. Given the need of a petrochemical complex to further strip the aldehyde (formaldehyde and acetaldehyde) to increase the quality of the MEG and increase the lifetime of the alpha-alumina-based silver catalyst, resin beds are designed and their surface absorption capacity is investigated to optimize aldehyde (formaldehyde and acetaldehyde) removal in the recirculating water flow of the ethylene oxide unit. Experiments show that the ion exchange system based on strong anionic resin pre-treated with a sodium bisulfite solution can reduce the aldehyde level from about 300ppm to less than 5ppm. After the resin is saturated with aldehyde, the resin can be recycled using the sodium bisulfite solution which is a cheap chemical substance.

Keywords: Bed anion resin, Ethylene glycol, Ethylene oxide, Formaldehyde, Return water

1 Introduction

MEG is a transparent, colourless, odourless liquid with a low volatility and is soluble in water. This substance is obtained from the reaction of ethylene oxide and water. The Shazand Petrochemical Company is an EG generation unit with an annual capacity of 105000 tons, located in the Markazi province of Iran (Figure 1). According to a global estimation, about 9.4 million tons of MEG generated in 1993 were used as an intermediate material in manufacturing polyester fibers, polymer film manufacturing industries, manufacturing bottles and packaging containers and so on [1]. Polyester fibers, polymer films and polyester resins are formed from the reaction of MEG with divalent acids and their esters. Due to a high mechanical resistance, polyester films are used in photographic films and magnetic tapes. They are widely used in packaging food materials with Polyethylene Terephthalate (PET), and also in the synthesis of polyethylene terephthalate of MEG, and also in the textile industry. Adding MEG to car's radiator water increases the boiling point of the water and decreases its point of freezing.

One of the problems of a generation line, is the destruction of the MEG at a high temperature, and in the presence of oxygen, it causes the formation of carboxylic acids (formic acid, acetic acid and so on) which can decrease the pH and cause corrosion of carbon steel tanks [2]. Therefore, it is recommended that the amount of dissolved oxygen for carbon steel systems be kept below 20ppm. Oxygen scavengers are used to keep the amount of dissolved oxygen low. The ethylene glycol wastage has a concentration of 500-1000 milligrams per liter of MEG. Other organic compounds are usually lower than 100ppm [3]. A major source of glycol wastage is generated from defrost excess fluids [4]. Wastewater treatment plants, consider a concentration of 1-5% as the maximum concentration for an effective microbial degradation with an acceptable oxygen demand [4]. There are many methods to recycle the ethylene glycol existing in the wastage of this industrial unit such as: reverse osmosis [5], membrane distillation [6], pervaporation [7], vacuum distillation [8], ozonization [9], use of activated carbon adsorption [10,11], aldehyde separation through stripping [12], and ion exchange [13]. These methods have some advantages and disadvantages, and the two methods with the best performances are ion exchange and aldehyde separation through stripping. In

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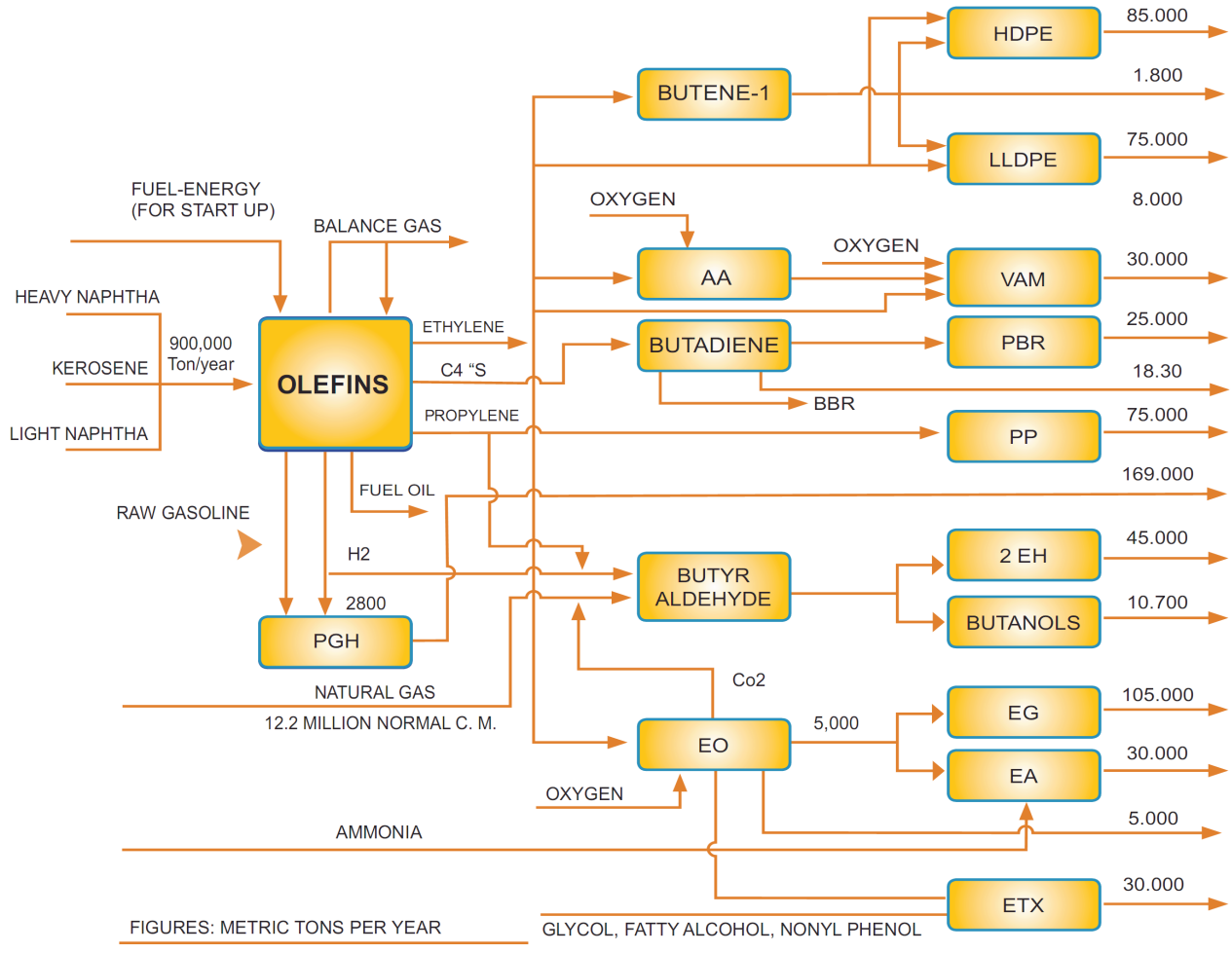


Figure 1: Block diagram of process unit of Shazand (Arak) petrochemical company.

this study, ion exchange with anion resin modified with sodium bisulfite were used to decrease the aldehyde and recirculate the wastage towards the ethylene glycol treatment unit [14,15] by Amberlite IRA 402C1 in 1atm and three different discharges.

2 Description of the process of producing ethylene glycols

Ethylene enters the ethylene oxide (EO) unit through a pipeline from the Olefin Plant (Figure 1) and the flow pressure is increased by the compressor and safely combined with the oxygen and recirculated gases and enters the reactor of a pipe which contains alpha-alumina-based silver catalyst. The oxidation temperature is controlled by the water in the reactor shell. The exhaust gases of the reactor contain N_2, CO_2, O_2, EO and aldehydes which enter the scrubber and the EO are absorbed in the form of a diluted aqueous solution. The unabsorbed exhaust gases are recirculated into the EO reactor after a heat exchange and pressure increase. A portion of the recirculating gas is sent to the stripping system before entering the reactor to strip the CO_2 which is stripped through a chemical reaction with a hot potassium carbonate solution of CO_2. Next, the liquid containing EO enters the stripper and the EO is stripped using some steam and the tower output flow is sent to purify the secondary stripper tower to strip light components such as aldehyde, water and so on from the final product. The stripper output water enters the recirculating water treatment system (weak anion resin transformer) after the removal of weak organic acids. The current level is 15-40 cm^3/hr. The treated recirculating water is used to adjust the temperature and prevent the glycol from leaving as the glycol evaporating reflux. Given

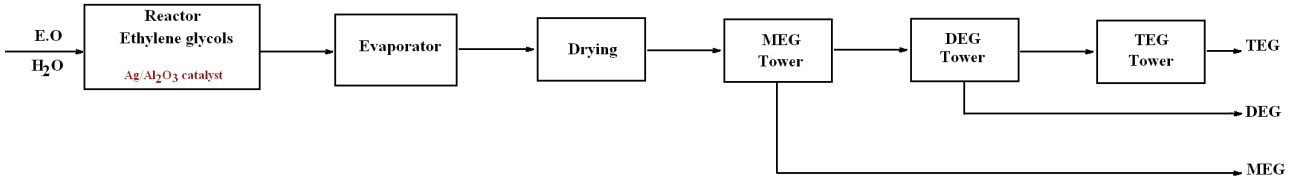


Figure 2: Production charts of ethylene glycol compounds.

the generation of aldehydes as an inappropriate side product in the process of ethylene oxide generation and the recirculation of the returned water, the aldehydes enter the final product which results in decreased purity of glycol products. It should be mentioned that with the usage of the catalysts, generation of side products such as aldehydes is increased which leads to increased returned water. If the amount of the aldehyde exceeds the limited amount, the returned water will be drained. Next, the aqueous solution current and ethylene oxide enter the EG unit reactor and MEG, DEG and TEG are generated at a specific pressure and temperature (Figure 2).

The output flow of the reactors, which contains about 10% ethylene glycol enters the series evaporator to strip the water flow from the product. The treated returned water is used as the reflux of the evaporators at this stage. Their chemical reaction is as follows (in 37~39 bar and 160~190 °C):

C_2H_4O (E.O) + H_2O → MEG [($C_2H_6O_2$) mono-ethylene glycol]
MEG + E.O → DEG [($C_4H_{10}O_3$) Diethylene glycol]
DEG + E.O → TEG [($C_6H_{14}O_4$) Triethylene glycol]

After that, the MEG, DEG and TEG products are obtained after passing through the series stripper towers. Based on the current process, the main problem is the existence of aldehydes in the returned water. According to existing standards, in the EO/EG unit, the amount of the existing aldehyde is 80ppm which, if increased to 120 ppm, the returned water is discharged to the wastage unit.

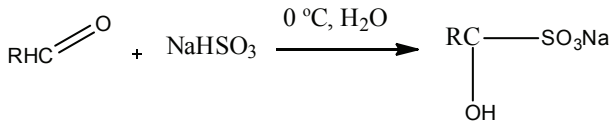
The purpose of the present project is to strip the aldehyde from the returned water of the unit to increase the purity of the glycol products. Therefore, one of the methods considered is to pass the returned water through a pre-treated strong anionic resin bed with a bisulfite solution.

3 Experimental methods

The items used in this study include sodium hydrogen sulfite, sulfuric acid, iodine, acetaldehyde, starch and soda made by the German company, Merck, and strong anionic resin of Amberlite IRA 402C1 made by the Rohm & Haas company. The tools and instruments required are gas chromatography equipment of Varian 3300-USA.

Determining the amount of aldehyde in the water samples of the ethylene oxide unit

Two flasks, one for the sample group and the other for the control group are considered. 50cc of distilled water is added to the control flask and 25cc of distilled water is added to the sample flask and 0.04N sodium bisulfite solution with an accuracy of 5.00cc is added to each flask. The two flasks are placed in a refrigerator for 10 to 20 minutes (maximum 30 minutes), next, both flasks are titrated using 0.01N of the iodine standard solution. The detection reagent for the equivalent point is starch adhesive. It should be noted that the reagent is added in the final stages of the titration. (At the beginning of the titration, the color of the added iodine quickly fades, but at the final stages of the titration, the added iodine is partially dispersed in the solution and it has a more stable color, after the titration reaches this stage, the starch adhesive solution is added. About one cc of the reagent is enough). The end of the titration is when the created blue color remains stable for at least 30 seconds. The volume of the iodine for the control and sample groups is registered. The important point is that the amount of the iodine used for the sample must never be less than 40% of the amount used for the control. In this case, the titration is repeated and this time, a greater amount of sodium hydrogen sulfite is added to each solution.



$NaHSO_3 + I_2O + H_2O \longrightarrow NaHSO_4 + 2HI$

when needed, the required amount of aldehyde can be calculated using one of the three formulas below:

$$\text{Aldehyde (As weight ppm of formaldehyde)} = \frac{(B-S)\times N\times 15000}{Vs\times Ds} \quad (1)$$

$$\text{Aldehyde (As weight ppm of acetaldehyde)} = \frac{(B-S)\times N\times 22000}{Vs\times Ds} \quad (2)$$

$$\text{Aldehyde (As weight ppm of acetaldehyde)} = \frac{(B-S)\times N\times 2.2}{Vs\times Ds} \quad (3)$$

Where B is the volume of the iodine used for the control group, S is the volume used in the iodine solution for the sample group, N is the normality of the iodine solution, V_s is the sample group's volume and D_s is the special weight (Sp.Gr) of the sample group. If it is required to report uncertainty, the value of the uncertainty is reported as follows: A± (0.019×A) where A is the amount of the aldehyde in the sample group.

Determine the amount of monoethylene glycol in water

First, one µl of the sample group is taken and placed inside the container of the gas chromatography equipment at a temperature of 240 °C, the sample is quickly evaporated and led towards the column using nitrogen gas (carrier gas to a pressure of 12 psi and discharge of 10 $ml.min^{-1}$) and then put inside the flame ionization detector's (FID) container. The existing column contains compressed polymer powder of polyethylene glycol with 20 moles of ethylene oxide (PEG, 20M), with a length of half a meter with a material type of stainless steel and with an 80/100 mesh. In the column is the stationary phase devices, it is selected of such which could affect on momoethylene glycol (MEG). The start of the temperature in the column is from 130°C which reaches 200°C after five minutes. At this temperature, water with a high peak (90%) appears, and after one minute, the peak of the MEG appears. To determine the weight percent of the MEG, the External standard method is used. The area under the curve (Area G.C) is calculated and using a response factor (RF) the concentration is calculated:

$$RF = \text{Area G. C} \Big/ \text{C(STD)} \quad (4)$$

Where C (STD) is a concentration obtained from the calibration of the equipment by the control sample.

Removal of aldehydes with strong anionic resin

In this method, a 4% weight MEG aqueous solution which contains an approximate amount of 300ppm of aldehyde (acetaldehyde) is passed through a strong anionic resin bed (Amberlite IRA 402Cl) pre-treated with bisulfite so the aldehyde is absorbed by the resin. An amount of 50cc of resin with water is poured in two burettes where the resin before coming into contact with the aqueous glycol solution, was first recycled with a 5% weight solution of NaOH to be changed into a resin-OH and then it was recycled twice with a 5% aqueous solution of sodium bisulfite to ensure bisulfite ion replacement according to the reaction below [16].

$$\text{Resin-OH}^-_{(S)} + NaHSO_{3(aq)} \rightarrow \text{R-HSO}_3^-{}_{(S)} + NaOH_{(aq)}$$

500cc of the acquired solution is poured into five 200cc beakers (each 100cc) and then using a dropper, it is slowly passed over the bed inside the burette. After the end of the reaction of each beaker, its aldehyde was measured to be below 5ppm. The passage of the solution over the bed continues until the aldehyde reaches 5ppm at the output. In higher amounts, the bed was prepared for recycling.

Bed reduction process

First 300cc of distilled water is passed over the bed using a dropper. According to the above description, first it was washed with a 5% weight soda solution to form the OH^- ions. Next, by preparing the 5% weight sodium bisulfite solution, the recycling stages take place. The method to prepare the sodium bisulfite solution is such that per each ppm of water-dissolved aldehyde, 3ppm of pure sodium bisulfite must be used, and since the maximum basis is considered to be 5ppm of aldehyde, therefore 15ppm of pure sodium bisulfite must be mixed with water to reach the desired volume. Next, using a dropper, it is slowly passed over the bed and collected at the exit, and the aldehyde amount is measured. If the amount of the aldehyde exceeds 5ppm, the passage of the bisulfite solution over the bed continues so at the end, the burette output reaches below 5ppm of aldehyde where all the recycling stages come to an end. Having recycled the sodium bisulfite, the resins were washed with enough deionized water.

$$\text{R-HSO}_3^-{}_{(S)} + HCHO_{(aq)} \longrightarrow \text{R-HOCH}_2\text{SO}_3^-{}_{(S)}$$

$$\text{R-HOCH}_2\text{SO}_3^-{}_{(S)} + NaHSO_{3(aq)} \longrightarrow$$

$$\longrightarrow NaHOCH_2SO_{3(aq)} + \text{R-HSO}_3^-{}_{(S)}$$

4 Results and discussion

The purpose of this operation is to reduce the pollutants inside the water obtained from the reaction of the generation of the ethylene oxide in the reactors. A flow of recirculating water in the ethylene glycol unit of Shazand Petrochemical Company with a discharge of about 20m^3/hr enters the treatment section, where 24 samples were randomly selected during different days of a year from the water going into the beds and coming out of their output to measure the amounts of the bed input and output aldehydes, MEG and ultra violet (U.V). Table 1, shows the values of these parameters which shows that due to the incomplete treatment of the water with a high aldehyde concentration and the output water, which contains MEG, it cannot return to the process as the reflux of the evaporating towers due to a low U.V and must be led towards the wastage of the unit. For example, in the fourth sample where the aldehyde is 128.9ppm, the U.V value has decreased. It should be mentioned that the high and low levels of the aldehyde values in table 1 is due to the fluctuating reaction conditions in the ethylene oxide reactors.

By designing the strong anionic resin bed pretreated with sodium bisulfite and passing the recirculating water through it, based on the samplings performed, acceptable results were obtained.. Also, in order to test the aldehyde stripping with the pretreated strong anionic resin, in three different discharges, including BV.hr (6, 8, 10) the solution will be passed through the prepared bed. The selection of these discharges is such that in industrial and laboratory scales according to the formulas of the company making the resin, the bed volume is selected based on the type of the resin and the amount of the bed input flow [17].

At each stage, the 500cc aqueous solution containing 4% weight of MEG and 300ppm aldehyde (equivalent to 10 times the resin volume) was passed through the bed. It should be mentioned that the test for measuring the aldehyde in the treated solution was performed in the laboratory of the EO/EG unit with Iodometry. After each stage of the test, the resin bed was recycled using a 5% bisulfite solution to keep the testing conditions fixed for each flow amount.

Table 1: Input aldehyde and ethylene glycol and the amount of UV light to enter the beds.

Sample	Date	UV/T%	MAG/wt.%	Aldehyde/ppm
1	24-03-2015	98.2	4.91	32.4
2	2015-04-04	94.1	6.02	91.9
3	2015- 04-30	95.8	4.71	70.0
4	2015-05-21	89.3	5.12	128.9
5	2015-06-05	94.2	3.48	47.3
6	2015-06-22	93.5	3.05	94.0
7	2015-07-07	91.0	4.85	96.4
8	2017-07-23	93.1	4.46	73.9
9	2015-08-02	87.2	5.00	101.4
10	2015-08-06	92.9	3.68	53.0
11	2015-08-23	92.2	3.58	43.0
12	2015-09-22	92.4	3.80	35.6
13	2015-10-07	88.1	3.36	111.2
14	2015-10-22	90.8	5.60	78.7
15	2015-11-06	91.3	4.75	52.0
16	2015-11-21	92.5	4.19	59.8
17	2015-12-06	90.1	4.27	61.3
18	2015-12-16	90.9	5.76	57.3
19	2015-11-26	92.6	5.01	48.3
20	2016-01-10	91.2	3.89	49.4
21	2016-02-04	90.2	9.10	50.6
22	2016-02-19	90.5	6.98	76.8
23	2016-03-05	90.8	3.74	74.8
24	2016-03-19	91.7	5.14	73.2

Aldehyde effects on UV of inlet water to anionic substrates

One way to assess the production mono-ethylene glycol is to measured its UV. For ethylene glycol with high quality, the UV is a high level (at least 95%), therefore when the aldehyde in the water cycle goes up, according to Figure 3, the UV of the water cycle goes down where the treatment load of the bed is increased and the use of the above water is not possible and should be sent to the wastage.

When the amount of the bed input aldehyde goes up, due to the increased treatment load in the bed, the aldehyde is not expected to decrease in the output water. In this state, the UV is also decreased and it has a direct effect on the quality of the MEG product. In the process of the generation of the MEG, the amount of the UV of the reactor output water is a very important factor in controlling the operational conditions.

At the end of the efficiency of the catalyst, the amount of the reactor output aldehyde reaches above 200ppm, where due to the saturation of the reactors with aldehyde and the lack of a decrease in its concentration of the output, the reactor input water is sent to the wastage. In Table 2, shows the amounts of the aldehyde and UV of output water from the anionic reactors. The reactor cannot decrease any more than 20-30ppm of the reactor input

Table 2: Input aldehyde and the amount of UV light passing out of the reactor.

Sample	Date	UV/T%	MAG/wt.%	Aldehyde/ppm
1	24-03-2015	99.10	4.89	9.40
2	2015-04-04	98.70	3.72	69.80
3	2015- 04-30	98.00	3.49	62.50
4	2015-05-21	93.40	8.71	110.10
5	2015-06-05	98.10	3.81	22.60
6	2015-06-22	96.50	5.74	78.30
7	2015-07-07	96.80	4.89	90.00
8	2017-07-23	94.00	4.21	59.90
9	2015-08-02	94.50	4.02	97.10
10	2015-08-06	97.50	6.88	30.20
11	2015-08-23	98.10	3.55	32.60
12	2015-09-22	97.30	3.47	29.80
13	2015-10-07	96.80	3.31	100.15
14	2015-10-22	96.90	4.55	55.70
15	2015-11-06	97.80	4.20	43.90
16	2015-11-21	96.80	3.62	37.90
17	2015-12-06	96.30	5.00	45.10
18	2015-12-16	97.70	4.69	51.10
19	2015-11-26	96.20	3.70	31.40
20	2016-01-10	97.90	3.00	23.60
21	2016-02-04	95.80	4.80	48.20
22	2016-02-19	96.00	6.00	70.60
23	2016-03-05	96.20	5.51	56.70
24	2016-03-19	96.90	5.00	62.90

aldehyde when the aldehyde is being increased. Therefore the above situation caused the pretreated anionic resin to be considered for decreasing the aldehyde.

In this state, the aldehyde changing curve on the UV is seen (Figure 4). The reason for the high prescence of the aldehyde in these samples is the high prescence in the reactor input. The results of sample number one, show some of the best reactor functioning conditions which, given the amount of its input aldehyde namely 32.4 ppm of Table 1, as the output its aldehyde reaches 9.4 ppm.

The effects of fluid flow

In this test, first passing 500cc of the sample (the samples were in five beakers and each were 100cc) in this volume of the resin, the amounts of the aldehyde were measured in five stages.

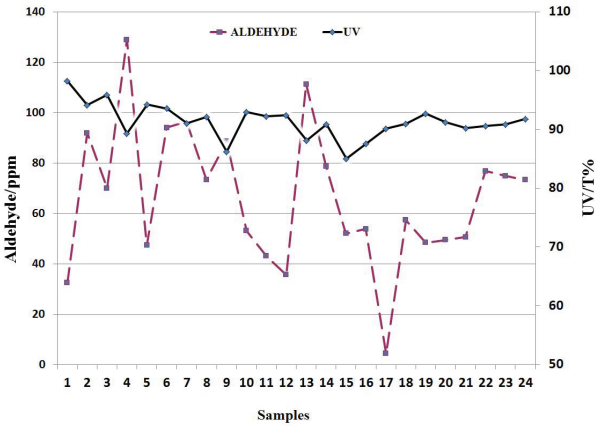


Figure 3: Aldehyde decreased with increasing the amount of water cycle entering to anionic reactor.

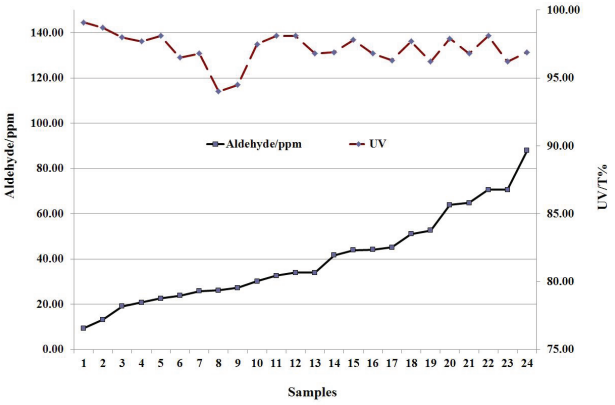


Figure 4: The effect of anion resin in ethylene glycol units, reducing the aldehyde in the water cycle output from the reactor.

After passing 100cc from the first beaker, the primary amount of 300 ppm per discharge 6BV.hr^{-1} (bed value per hour) reaches 1.9 ppm for a volume of 100cc, which with increased liquid discharge to 8BV.hr^{-1}, the amount of the aldehyde was decreased and again with the increase of the discharge to 10BV.hr^{-1}, it shows a significant increase.

In subsequent volumes of the solution passage, at a discharge of 6BV.hr^{-1}, the amount of the aldehyde was fixed and at the end of the final stage, its amount increased to 1.7 ppm, but for a discharge of 8BV.hr^{-1}, in the passage of the solution over the pretreated strong anionic resin bed, the amount of the aldehyde was significant decreased with a little fluctuation. The results from the bed discharge changes can be seen in Table 3.

In other words, it must be said that the concentration of the aldehyde after the passage of about 100cc of the solution, quickly decreased from the primary 290ppm to 300ppm in the 4% MEG solution to less than 2ppm in discharges of 6BV.hr^{-1} and 8BV.hr^{-1}, which shows the high capacity of the resins pretreated with bisulfite and the appropriateness of the ion exchange method in the

Table 3: Results bed flow and its impact on reducing the aldehyde concentration from 300 ppm initial concentrations.

V/cc	Concentration/ppm		
	6 /BV.hr^{-1}	8 /BV.hr^{-1}	10 /BV.hr^{-1}
100	1.92	1.48	3.50
200	1.50	1.45	3.90
280	1.50	1.22	3.90
380	1.50	1.39	3.90
500	1.70	1.22	3.10

Table 4: The volume of samples 8BV.hr^{-1} compared the UV output from the reactor.

V/cc	UV/T%
110	95.50
180	95.39
250	95.31
300	95.25
370	95.00
425	95.20
500	95.20

aldehyde stripping. Also based on the diagram, it is observed that the amount of the stripping of the aldehyde in discharges of less than 10BV.hr^{-1} was performed much better which shows a higher time for the transfer of mass and a better aldehyde stripping. It should be mentioned that for aldehyde concentrations of less than 5ppm, the test results have errors and in general, the lower values are reported as a trace. Therefore, the better results were obtained in the 8BV. hr^{-1} discharge, therefore in different volumes of this sample, the amounts of UV were measured. In table 4, the values of the sample volumes passing through the bed with a discharge of 8BV.hr^{-1} and the measurement of their UV amounts are compared.

According to table 4, is showing the amount of the passage of the UV from the treated solution. This amount decreased from 95.5 at a wavelength of 275nm for the primary solution to about 95 in the treated sample.

5 Conclusion

Based on the objective achieved in this study, it can be concluded that the recirculating water cycle of the EO/EG unit of Shazand Petrochemical Company, after passing through weak anionic beds, does not have the ability to decrease the amounts of aldehyde any more than 40ppm and cannot increase the UV of the output water to above 95 (minimum basis for designing the purity of the UV of the MEG product). Therefore, in the study performed, the use of an ion exchange system was investigated. The tests show that the ion exchange system based on a strong anionic resin of Amberlite IRA 402C1 pretreated with a sodium bisulfite solution can decrease the amount of the aldehyde from about 300ppm to less than 5ppm. After the saturation of the resin with aldehyde, the resin can be recycled using a sodium bisulfite solution which is an inexpensive chemical substance. It should be mentioned that given the high capacity of the resins, after the passage of 8BV/hr of the solution, the concentration of the aldehyde remains around 2ppm. It is recommended to continue experimenting and properly design a process on a pilot scale (with at least 50L of resin at an appropriate ion exchange column along with all the injection and stationary equipment).

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