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## **THE DEPENDENCY ON THE TEMPERATURE OF EFFICIENCY OF THE REGENERATION PROCESS IN GLYCOLS\*\*\***

### **1. INTRODUCTION**

Mined natural gas deposits, in addition to valuable components such as methane, ethane, propane, butane and other hydrocarbons, also contain impurities. Redundant components, including water, nitrogen, carbon dioxide and hydrogen sulfide, must be removed before submitting the gas to public transmission and distribution pipelines or before condensation, to LNG, in order to meet the quality requirements set forth by Polish law and standards.

The most common impurity in natural gas is water, which may be in the form of steam, condensate (as a result of gas expansion) and in solid form (ice, hydrates). The biggest technological problems are caused by the last two items listed. Water, in liquid form, encourages the corrosion of materials. Distorts the measurement results of gas flow intensity. Whereas the hydrates cause damage to the system, especially to measuring devices and fittings, while they accumulate on the walls of the pipeline, causing a reduction in its capacity [8]. Therefore, natural gas is subjected to the dehumidification (removal of water) process, which can be divided into:

- adsorptive,
- absorptive,
- expansive,
- membranous.

The widest application used in dehumidifying natural gas has found methods of absorption, which rely on the passing of moist gas through a highly hygroscopic medium.

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Such mediums (absorbers) include the glycols listed below [14]:

- ethylene – EG ( $C_2H_6O_2$ ),
- diethylene – DEG ( $C_4H_{10}O_3$ ),
- triethylene – TEG ( $C_6H_{14}O_4$ ),
- tetraethylene – TREG ( $C_8H_{18}O_5$ ).

Currently, the most used triethylene glycol (TEG), which thus far displaces the used diethylene glycol (DEG). The choice of the method in dehumidifying depends on many factors, however, the most important ones include the flow rate and intended use of the dehumidified gas. Compared to adsorption, the regeneration method that uses glycols has the following advantages:

- lower cost of installation – for lower process efficiency, the savings on construction is greater,
- a smaller drop in natural gas pressure (0.7 bar compared to 3.5 bar),
- the process is continuous,
- supplementing glycol does not require a break in the installation,
- absorption plant operating costs are lower – glycols have lower heat capacity than the solid adsorbents, resulting in less heat to be supplied for regeneration,
- a high degree of dehumidification of natural gas, up to 99.9% for assisting the process by gas stripping.

The disadvantages of the absorption dehumidifying process of the natural gas can include:

- dew point temperature below  $-30^{\circ}C$  requires the use of stripping gas installations,
- glycols are susceptible to contamination (impurities),
- glycols cause corrosion of the system, if they are contaminated or if there is a distribution of glycol due to overheating.

During the dehumidifying process of natural gas the glycol absorbs water (usually a change of concentration of a few percent) and for the glycol to be able to keep the original sorption properties, it should undergo a process of regeneration, consisting in removing the water by thermal evaporation. The regeneration process is possible thanks to the large difference between the water vapour pressure (more than 105 Pa/ $100^{\circ}C$ ) and glycol (about 200 Pa/ $100^{\circ}C$ ) and the high temperature of glycol evaporation. However, the fact that the glycol has a lower thermal decomposition temperature than the boiling point causes difficulties in regeneration [17].

## 2. METHODS OF GLYCOL REGENERATION

Regeneration of glycol is the stage in which the dehumidification process of the natural gas allows for the restoration of its original water absorbency. The most common method of regeneration used to date is the thermal evaporation of glycol at atmospheric pressure. The process relies on the distilling of the mixture in a regenerator composed in a boiler (where the glycol is heated) and the distillation columns (wherein the separation of water). Watered glycol is directed from the absorption column to the coils located at the top part of the distillation column, and then to a degasser, where separation of the gas from glycol occurs. The glycol

is directed further through the filter and into the boiler where it is heated to remove residual water. Subsequently, with the use of a pump, it is fed once again to the absorption column. The disadvantage of this solution is the emission of pollutants dissolved in water vapour from the regenerator column into the atmosphere, in which:

- natural gas derived from incomplete degassing of glycol,
- methanol vapour,
- decomposition products of glycol,
- aromatic hydrocarbons contained in natural gas.

Another disadvantage of this process is the thermal decomposition of the glycol [17].

After the standard regeneration, the solution typically contains up to 98.7% of glycol, which may obtain a dehumidified gas temperature (10°C to 25°C) a dew point in the range of 2°C to –28°C. These values are sufficient for the transmission network. The achievement of lower water content in natural gas requires the use of modified methods of regeneration which lead to a very high concentrations of glycol.

One way of obtaining a higher concentration of glycol is to reduce the partial pressure  $H_2O$  in the regenerator boiler. In order to achieve this effect, the purging gas from pre-generated glycol (stripping gas) is used. The purging gas is obtained mostly from the glycol degasser and is heated in the coil, forming part of the regeneration column. It can be fed into the stripping column [17].

Another method of glycol regeneration which results in a very high concentration of glycol is “Drizo®” type regeneration. It is a combination of azeotropic distillation (natural gas containing certain amounts of heavier hydrocarbons) with stripping. Dump gas from the regenerator is not directed to the atmosphere, but cooled and directed to a separator, where the separation of water from the higher hydrocarbons occurs. These hydrocarbons are used later to increase the efficiency of the regeneration of glycol.

Increase in productivity of the glycol regeneration is also achieved by the combined use of gas stripping and vacuum pumps. The process involves the formation of negative pressure in the regenerator column by pumping out the vapour and gas.

Enhancement of the glycol concentration can also be obtained by condensation of water vapour in a specially prepared installation. A bundle of condensation tubes is mounted within the steam tank overflow. A cooling medium flows through the capacitor (hence the name of this method – “Coldfinger”), and the difference in temperatures results in condensation of water vapour.

The current regeneration methods utilise the heat coming from outside (on the principle of convection) to heat the glycol. This type of process has two fundamental disadvantages:

- 1) glycol is heated unevenly (this may result in hotspots), whereby part of the glycol is not suitable for dehumidifying the gas and must be replaced,
- 2) the long regeneration process is due to the large size of the columns.

These disadvantages necessitate the search for new construction solutions for regenerators, with the aim to improve the efficiency of glycol regeneration. One of the research directions is the use of microwave radiation energy in regeneration [8]. The process involves converting the electromagnetic energy into thermal energy in a given substance and takes place directly in the entire glycol volume. Thanks to this; the speed of reconstruction can be significantly increased while decreasing the loss of glycol, compared to the process of

convective evaporation. Microwave radiation does not cause the breaking of glycol's chemical bonds due to the low power of the radiation.

Microwaves are one type of electromagnetic radiation with a wavelength from 1 mm to 1 m (corresponding to frequencies from 300 MHz to 300 GHz). This range is located between the infrared and ultra-short waves. In Europe, the most commonly used frequency for industrial, scientific and medical applications is 2.45 GHz due to the reduction of noise in telecommunications.

The energy of microwave radiation is absorbed into substances by three mechanisms:

1. magnetic losses in ferromagnetic materials,
2. ohmic losses in conductive materials,
3. electric losses caused by electromagnetic non-uniformity (e.g. ions or molecules dipoles).

Absorption of radiation into propylene glycol is conducted by the third mechanism, the so-called polarisation of dipoles. As a result of the action of the electromagnetic field, the chemical molecules (dipoles) are trying to set themselves in accordance with the direction and the reversal of this field which changes their arrangement. This change causes the collision between adjacent molecules and their subsequent exchange of radiation energy. These in turn collide with the following molecules, which results in an increase in thermal energy of glycol in the entire volume in a coordinated way [1, 15, 16].

### 3. GLYCOLS USED IN THE GAS DEHUMIDIFICATION PROCESS

Many substances have the ability to absorb water. However, only a few have been used as absorbents in the gas dehumidification process, because in addition to high hygroscopicity they must also be characterised by:

- the lack of impact of corrosion on system components,
- low affinity for the components of natural gas (the lack of chemical reactivity),
- thermal stability, particularly at high-temperature changes in the heat exchanger,
- ease of regeneration to a high concentration, so that after removing water from it, it can be reused in the process of gas dehumidification,
- low viscosity,
- low vapor pressure, to minimise the loss of absorbent,
- low solubility in hydrocarbons,
- low tendency to foaming and emulsification.

In addition, an important factor is the economic aspects.

In practice, it is possible to apply the glycols listed below in dehumidifying natural gas:

- ethylene glycol (EG),
- diethylene glycol (DEG),
- triethylene glycol (TEG),
- tetraethylene glycol (TREG).

The heavier glycols (TEG and TREG) are more hygroscopic than the lighter ones (EG and DEG). Moreover, heavier glycols, compared to the EG and DEG, have a lower vapour pressure.

TEG and TREG regenerate easily because they have higher thermal stability and a lower affinity for water. TREG possesses the highest viscosity of the above-mentioned glycols – and the higher the viscosity of glycol, the greater the costs to be borne for its distribution in the system. On the other hand, TREG possesses the most advantageous properties for dehumidification and regeneration of the glycols mentioned above, however, the economic aspect (TREG is the most expensive), and comparable properties have determined that currently TEG, has the widest application [4, 14].

The advantages of triethylene glycol as compared to the formerly used lighter glycols include:

- TEG is easier to regenerate at high concentrations of up to 98–99.95% at atmospheric pressure since its boiling point is much higher – high concentrations of glycol are important from the point of view of the gas dehumidification since the dew point temperature for the natural gas is lower then (the process is then more efficient),
- TEG has a much higher decomposition temperature, which is advantageous for the regeneration process because of the lower risk of overheating and a higher concentration – for comparison the decomposition of TEG occurs at 207°C, and DEG – at 164°C.
- loss of TEG in the regeneration process (evaporation of water) is smaller due to the lower vapour pressure,
- the total operating costs for use of TEG are lower,
- the viscosity of TEG (above room temperature) does not impede the process of distribution at the installation of dehumidification and regeneration [9].

### Selected properties of glycols

Glycols are colourless and odourless liquids. Compared to water, glycols have a higher boiling point, a lower freezing point and a lower vapour pressure. Furthermore, they have a higher viscosity and are slightly heavier. The solubility of glycol in water is unlimited, while in other alcohols, ketones and aldehydes it is very good. The solubility of the glycol in aliphatic hydrocarbons is very poor.

The selected technological and thermodynamic parameters of glycols are shown in Table 1.

**Table 1**  
Basic thermodynamic parameters of glycols used to dehumidify gas [3]

Parameter	Unit	EG	DEG	TEG	TREG
		C2H6O2	C4H10O3	C6H14O4	C8H18O5
Boiling point at 1 atm.	[°C]	197.1	244.7	287.8	304.9
Melting point at 1 atm.	[°C]	-12.60	-7.80	-4.30	-4.10
Decomposition point	[°C]	165	164	207	238
Density at 25°C	[g/ml]	1.111	1.114	1.119	1.122
Density at 60°C	[g/ml]	1.085	1.089	1.092	1.096
Freezing point	[°C]	-13.4	-8.7	-7.2	-4.1
Viscosity at 25°C	[cP]	14.06	30.42	31.43	33.66
Viscosity at 60°C	[cP]	5.00	8.92	9.93	9.94

**Table 1 cont.**

Parameter	Unit	EG	DEG	TEG	TREG
		C2H6O2	C4H10O3	C6H14O4	C8H18O5
Surface tension at 25°C	[dyn/cm]	47.98	48.09	44.99	44.00
Heat of vaporization at 25°C	[cal/g]	197.04	73.55	81.69	99.18
Specific heat at 25°C	[cal/g·C]	0.571	0.559	0.500	0.562
Heat of fusion	[cal/g]	38.34	36.94	49.65	45.01
Heat of combustion	[MJ/m <sup>3</sup> ]	126.2	237.2	495.0	495.0
Flash point	[°C]	115.5	137.8	160.0	204.0
Autoignition point	[°C]	410	370	370	358
Lower explosive limit	[% vol.]	3.2	2.0	0.9	0.5
Upper explosive limit	[% vol.]	53.0	22.0	9.2	3.4

An important parameter for the process of regeneration of glycols is their decomposition temperature. Due to the fact that it is lower than the boiling point temperature of glycols, it determines the maximum temperature of the process [6].

#### 4. LABORATORY TESTS

##### The scope and purpose of the tests

Within the framework of the laboratory tests, the measurements of the glycol solution's dehydration were conducted with the use of convection heating for the test, a series of several test solutions were prepared: EG, DEG, TEG and TREG at a concentration of 10% weights. Measurements were carried out under different temperature conditions and at the same heating times, while using a vacuum pump to evaporate the water vapour, were generated (except for one of the test series). The decomposition temperature of the EG was adopted as the upper-temperature limit, due to its lowest value compared with heavier glycols.

The efficiency of regeneration was evaluated on the basis of the analysis of water content in the samples after the heating process. The water content in the solutions was determined by the Karl Fischer titration method.

##### A methodology for measurement of the water content of glycols

The equipment of laboratory KIG WWNiG AGH includes volumeter (volumetric titrator) DL38 Mettler-Toledo, used for rapid and accurate analysis in the range of several hundred ppm to a 100% water content in liquids (Fig. 1). It is based on the use of Karl Fischer's methodology, which belongs to the chemical methods in measuring water content.

It involves the direct titration of the test solution of Fischer reagent (methanolic solution of iodine, sulfur oxide (IV) and pyridine) and calculation of the percentage of water based on the volume of the Fischer reagent consumed during the titration. The principle of the method consists in the chemical reaction of water, present in the sample with a solution of iodine and sulfur dioxide in methanol/pyridine (Karl Fischer reagent), that occurs during titration:





**Fig. 1.** Volumetric titrator DL38 Mettler-Toledo in laboratory KIG WwNiG AGH

The Karl Fischer methodology also allows for the coulometric determination of water content, where the mass of a substance is determined on the basis of the measured electrical charge required to change the oxidation state of the substance to be determined. The Mettler-Toledo Coulometer provides the determination of water content in the range of 1 ppm to 5%.

The Karl Fischer method is suitable for the determination of free water in most liquid chemical products, both organic and inorganic.

The analyses performed by automatic titrators have many advantages, such as improved reproducibility of results, the simplification of the measurement procedure, speed of action and the high accuracy of the determination of water content.

### Preparation of samples

A series of six tests were prepared – each consisting of four samples of an aqueous solution of glycol: ethylene, diethylene, triethylene and tetraethylene of 10% weights – that are heated at different temperatures. Samples were prepared by weighing 90 g of glycol and by adding 10 g semi-automatic pipette of water.

Subsequently, the concentration of the resulting solutions (Tab. 2).

**Table 2**

The water content of the samples of the all series of testing before heating

Aqueous solution	Mass concentration [%]					
	I series	II series	III series	IV series	V series	VI series
EG	10.09	9.94	10.00	9.93	10.02	9.98
DEG	9.97	9.99	10.06	10.00	9.94	9.97
TEG	9.99	9.97	10.11	9.98	10.01	9.98
TREG	9.94	9.93	9.97	9.99	10.05	9.98



Before placing in the chamber, the samples of watered glycols were mixed together. For this purpose, a device from FALC Instruments was used.

### Heating of samples

Each of the prepared series of solutions was poured into a teflon container, closed and placed in the heating chamber in order to be heated at temperatures consecutively 128°C, 133°C, 138°C, 143°C and 148°C during approx. 2.5 hours counted together with the time it took to reach that temperature. After reaching the predetermined temperature, samples were cooled together with the furnace. The containers in the heating chamber are connected to each tube in order to empty out the water vapour during cooling. For this purpose, a KNF NSE800 vacuum pump was used (Fig. 2).



Fig. 2. Containers with samples in the heating chamber

Additionally, one of the series of tests was heated at a temperature of 143°C without removing the vapour formed during regeneration of glycol – regeneration was performed at atmospheric pressure.

### The measurement of water content

The water content in the samples was measured by a DL38 Mettler-Toledo volumetric titrator immediately after cooling the samples from the heating chamber. For each sample, three measurements were made and the results averaged.



## 5. RESULTS

The first series of samples of glycol was heated to 128°C and further to a temperature 5°C higher. An exception was the last one, the sixth series of the test, which was heated to 143°C, just as the fourth series of samples. In this case, the process was not aided by a vacuum pump.

After cooling down titrated 0.2 g to 0.25 g of each type of glycol. For each sample, three tests were performed. Test results of water amounts are summarised in Tables 3–7.

**Table 3**

The water content of the first series of samples after being heated at the temperature 128°C

I series			
Aqueous solution	Mass concentration [%]		
EG	9.52	9.48	9.45
DEG	8.40	8.30	8.38
TEG	7.78	7.69	7.69
TREG	7.08	7.15	6.96

**Table 4**

The water content of the first series of samples after being heated at the temperature 133°C

II series			
Aqueous solution	Mass concentration [%]		
EG	9.00	8.86	8.69
DEG	7.40	7.35	7.42
TEG	6.62	6.55	6.59
TREG	6.03	6.08	6.10

**Table 5**

The water content of the first series of samples after being heated at the temperature 138°C

III series			
Aqueous solution	Mass concentration [%]		
EG	8.34	8.36	8.36
DEG	6.50	6.45	6.58
TEG	5.49	5.45	5.44
TREG	4.73	4.75	4.77

**Table 6**

The water content of the first series of samples after being heated at the temperature 143°C

IV series			
Aqueous solution	Mass concentration [%]		
EG	6.34	6.35	6.28
DEG	4.51	4.52	4.50
TEG	3.66	3.71	3.73
TREG	3.08	3.19	3.20

**Table 7**

The water content of the first series of samples after being heated at the temperature 148°C

V series			
Aqueous solution	Mass concentration [%]		
EG	5.64	5.64	5.65
DEG	3.94	3.94	3.96
TEG	3.03	3.05	3.03
TREG	2.71	2.74	2.74

Regeneration effects of specimens were evaluated based on the water concentration difference before and after regeneration. The efficiency of the process has also been calculated.

**Table 8**

Dewatering process parameters of glycols solutions

Heating time [h]	Temperature [°C]	Water content after process [% weight]	Differences in water concentration [% weight]	Process efficiency [%]
Ethylene glycol (EG)				
2.5	128	9.48	-0.61	6.02
2.5	133	8.85	-1.09	10.97
2.5	138	8.35	-1.65	16.46
2.5	143	6.32	-3.61	36.35
2.5	148	5.64	-4.38	43.72
2.5*	143	9.07	-0.91	9.15

**Table 8 cont.**

Diethylene glycol (DEG)				
2.5	128	8.36	-1.61	16.16
2.5	133	7.39	-2.60	26.01
2.5	138	6.51	-3.55	35.29
2.5	143	4.51	-5.49	54.88
2.5	148	3.95	-5.99	60.26
2.5*	143	7.23	-2.74	27.51
Triethylene glycol (TEG)				
2.5	128	7.72	-2.27	22.71
2.5	133	6.59	-3.38	33.90
2.5	138	5.46	-4.65	46.01
2.5	143	3.70	-6.28	62.93
2.5	148	3.04	-6.97	69.62
2.5*	143	6.08	-3.90	39.08
Tetraethylene glycol (TREG)				
2.5	128	7.06	-2.88	28.95
2.5	133	6.07	-3.86	38.85
2.5	138	4.75	-5.22	52.36
2.5	143	3.16	-6.83	68.37
2.5	148	2.73	-7.32	72.8
2.5*	143	5.41	-4.57	45.80

\* without pumping out the vapour during the regeneration process

The results summarised above (Tab. 8), show two principle patterns. The higher the homologous series of the glycol, the easier it regenerates. The water content of TREG is the lowest, and the largest for EG in spite of the same parameters of temperature and pressure of the process. Lowering the pressure during the process increases its efficiency. Although the samples were heated to the same temperature (143°C), escaping vapours formed during regeneration and therefore using pressure conditions close to a vacuum, reduces the water content of the glycol. Importantly, each measurement result confirms the two rules observed.

The lowest concentration of water is obtained for tetraethylene glycol at 148°C. This temperature is significantly lower than the decomposition temperature of the glycol. Slightly poorer results were obtained for triethylene glycol. The differences in process efficiency between the range from 4% to 7% depending on the process temperature (is greater if the difference in water concentration in glycols is lower). For ethylene glycol and diethylene, regeneration efficiency is considerably lower, provided that the first of the above is substantially different from the other (from 37% to 47% compared to TREG).

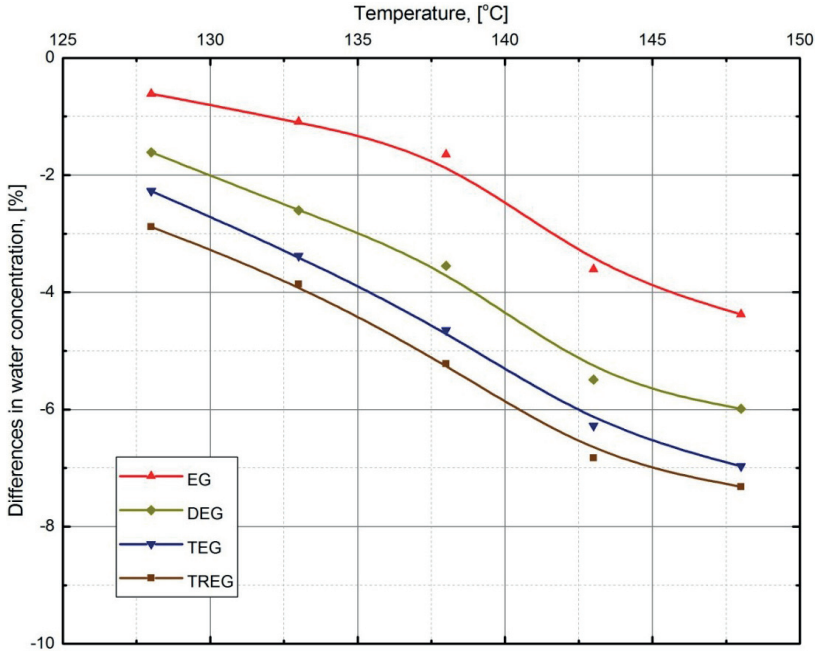


Fig. 3. The process of the differences in water concentration in glycols as a dependant on temperature

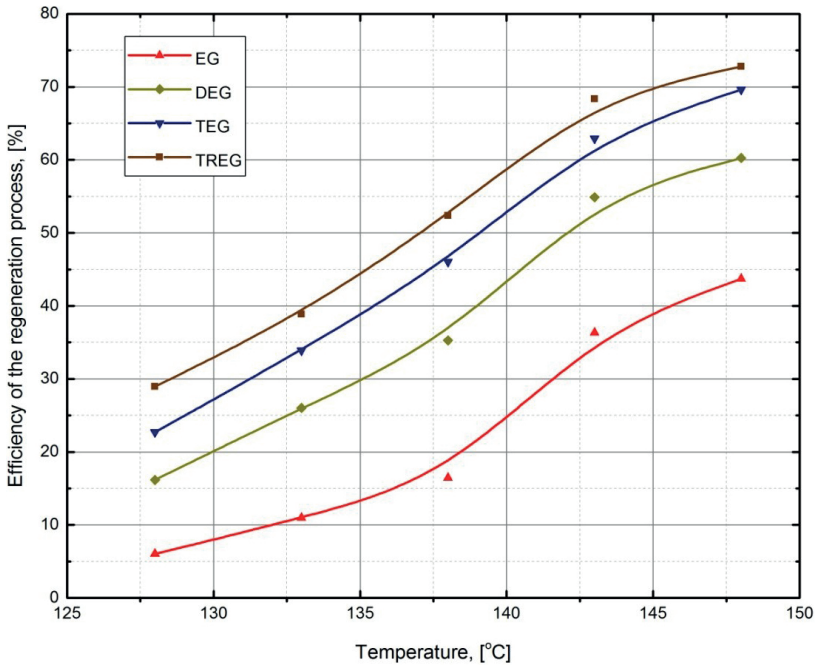


Fig. 4. The dependency on the temperature of efficiency of the regeneration process in glycols

The results are plotted in two graphs – the first showing the difference in water concentration in glycols as a function of temperature (Fig. 3), and the second showing the efficiency of the regeneration process in glycols at a fixed temperature (Fig. 4).

## 6. CONCLUSIONS

The above described test showed that:

- 1) Glycols: triethylene and tetraethylene have shown better parameters of the process compared to glycols: ethylene glycol and diethylene glycol. Their regeneration under the same conditions made it possible to obtain a higher concentration. This is mainly due to two factors. The higher homologues of glycol have a lower vapour pressure, thus the partial pressure difference between water and glycol is greater. This is advantageous from the viewpoint of the efficiency of regeneration. Moreover, they have a lower affinity of water – water remains in the non-bound state due to the smaller number of bonds in the molecule, which can be broken down by water.
- 2) Higher concentrations of glycol are provided at a higher temperature of the regeneration process. However, above 143°C the glycol regeneration efficiency improvement is no longer as noticeable.
- 3) Lowering the pressure during the regeneration has a positive effect on the concentration of glycol. The use of the vacuum pump has significantly improved the efficiency of the process and made it possible to obtain a concentration of 97.27% weights. For TREG at the temperature 148°C, the difference in water content of the glycol indicates that it is preferable to limit the temperature of the process, and apply methods of the process support such as purge gas stripping or using a vacuum pump.
- 4) The concentration of water in the TEG and TREG for the highest applied temperature of regeneration is similar. These glycols therefore, have parameters of the process that are similar at higher temperatures.

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