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Influence of ETBE addition on water tolerance of ethanol-gasoline blends

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Abstract: *Currently, there is a tendency for oxygenates fuels that can be produced from renewable sources. The widely used renewable fuel extenders are ethanol (100% renewable) and ETBE (47% renewable). The addition of renewable fuels in commercial fuels should be done carefully. The European Standards have modified in order to support the introduction of renewable fuels in commercial fuels. Specifically, the last revision of the Directive EN 228 standard (EN 228:2012) for gasoline resulted in the increase of the oxygen content at 3.7% m/m. In the present work the water tolerance of oxygenated ethanol-ETBE- gasoline blends was investigated. The results showed that the water tolerance was affected by the composition of the gasoline. ETBE is recommended as a co-solvent which enhances the retention of ethanol and water into the hydrocarbon phase improving their solubility, without phase separation. The higher water retention was recorded for the common presence of ethanol and ETBE in the gasoline. Three commercial gasolines were tested. Two of the three gasoline samples tested showed that the better water tolerance was achieved for the blends with a high content in both ethanol and ETBE. The third gasoline, with lower aromatic content and higher olefinic content, presented the better water tolerance for the lower ETBE addition (5% and 10% v/v).*

Keywords: *Gasoline, ETBE, ethanol, refinery blending components, water tolerance*

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I. Introduction

In the recent, there is intensive awareness about the climate change, air pollution, increasing energy consumption, and the discussions regarding shrinking oil supplies. The transport sector accounts for a third of the total energy consumption in the European Union, which depends mainly on imported crude oil. The Renewable Energy Directive establishes an overall policy for the production and promotion of energy from renewable sources in the EU. It requires the EU to fulfill at least 20% of its total energy needs with renewables by 2020. All EU countries must also ensure that at least 10% of their transport fuels come from renewable sources by 2020. Bioethanol is the most common biocomponent that can be used in motor gasoline. [1, 2] The addition of renewable fuels in commercial fuels should be done carefully, since the blend properties are altered, and, the engine efficiency may be reduced. The adoption of the new Directives resulted in the increase of the oxygen content at 3.7% m/m in the last revision of the EN 228 standard (EN 228:2012). [3, 4]

Alcohols as ethanol have been added to gasoline as octane boosters, oxygenators, a means of extending the fuel supply, and replacing ethers such as methyl tertiary butyl ether (MTBE). MTBE is the main oxygenate compound that is added in commercial gasoline. MTBE has a limited effect on the volatility of gasoline contributing to a high octane number. However, the MTBE-blended fuel should be restricted due to its toxicity and contamination of groundwater. [5] At present, there is a tendency for oxygenates fuels that can be produced from renewable sources instead of fossil fuels. The widely used renewable fuel extenders are ethanol (100% renewable) and ETBE (47% renewable). Fuel regulations are being adjusted in order to permit the addition of renewable components. From the refinery standpoint, the cost for revamping an existing MTBE unit to produce ETBE is very low, so it may be fungible. [6]

There is an issue with alcohols as ethanol. Water and alcohol are fully miscible and this can cause the alcohol and water to separate from the gasoline and form two distinct layers. The water in gasoline, in solution or a separate phase, can have different effects on an engine, depending on the type of engine being used. In case of separate water phases in the fuel, this can be damaging to an engine, while small amounts of water in solution with gasoline should have no adverse effects on engine components. The creation of a separate layer is termed phase separation. The point of phase separation is controlled by several factors: the type of alcohol, the amount of alcohol, the presence of co-solvents, the temperature, the hydrocarbon composition of the gasoline, and the amount of water. [7] During phase separation, the alcohol is partially removed from the gasoline forming a denser bottom layer composed of alcohol, water, and alcohol-soluble hydrocarbons. The upper less dense layer is gasoline rich partially depleted of alcohol and alcohol-soluble hydrocarbons. [8] This presents two problems.

The first is the possibility that the bottom layer can be stirred up and pumped into a vehicle and stall the engine. The second is that the upper layer will be reduced in octane value and may no longer meet specifications. [7]

Moreover, the addition of ethanol to gasoline affects the crucial physical-chemical properties of gasoline: volatility (as expressed by vapor pressure and distillation properties), octane number, water solubility, compatibility with construction materials, heating value. The vapor pressure of ethanol-gasoline blends is increased by ethanol addition (up to 10% v/v), altering gasoline performance characteristics and gasoline storage behavior. The distillation characteristics may also be altered by the formation of minimum boiling azeotropes between ethanol and hydrocarbons of gasoline. [9] On the other side, a higher ethanol addition (>10% v/v) forces the hydrogen bonds between ethanol molecules, resulting in a decrease of volatility. [10] Also, oxygenates have lower heating values than hydrocarbons on both unit-mass and unit-volume bases. The addition of oxygenates as ethanol to conventional gasoline decreases the heating value. The amount of this decrease depends on the amount and the identities of the oxygenated components used. [11] On the other side, the use of ETBE is beneficial since inhibits the elevation of gasoline volatility caused by ethanol addition. ETBE acts as a stabilizer enhancing the addition of lighter costless products as butane in gasoline composition. [10, 12] Also, the use of azeotropic mixture ethanol/ETBE is suggested in published works since it is environmentally attractive reducing the carbon dioxide emissions by the important replacement of gasoline. [13] Another advantage of ethanol and ETBE utilization is that the octane rating of gasoline is significantly increased with the addition of oxygenated compounds. [14]

The water tolerance in fuel blends is defined as the volume percentage of water that a blend can retain in solution, "tolerate" at a given temperature without phase separation. It is measured by adding water in small quantities by means of a burette to gasoline ethanol blends of known composition. Not all types of gasoline have the same tendency to retain water. The polar character of the ethanol due to the presence of the hydroxyl group is the main reason for the high affinity of ethanol with water. [11] Ethanol significantly improves the solubility of water in hydrocarbon fuel something which is useful and desirable due to the prevention of fuel system freezing. As referred in published works, when phase separation occurs somewhere between 30 to 70 percent of the ethanol will drop to the bottom of the tank with the water by changing ethanol concentration as the two layers come to an equilibrium. [15]

The composition of the gasoline is an important parameter that affects the water solubility and the separation of water from the gasoline. The composition of gasoline is a significant parameter since the aromatic hydrocarbons are characterized as the more polar hydrocarbons due to the presence of π bonds in the molecule. This more polar character has been found to increase the amount of water that can remain in a stable hydrocarbon – ethanol blend without phase separation. [8, 16, 17] Thus, according to the bibliography, the phase separation consists of an upper gasoline-rich phase mainly contains paraffinic hydrocarbons and a lower dilute aqueous ethanol-rich phase that contains some aromatic hydrocarbons that are soluble in ethanol.

The use of oxygenated gasoline demands the investigation of the use of some oxygen containing compounds as co-solvents to improve the phase stability of gasoline-ethanol blends (reduction of phase separation temperature). The main function of co-solvent is to enhance the overall polarity of fuel so that ethanol and water are drawn into the system. Once ethanol and water are in the system, then the stabilizing additive solubilizes these compounds. As highlighted in published works, ETBE as a co-solvent could benefit oxygenated gasolines since it can be blended into gasoline with the aim of improving the solubility of water in gasoline-ethanol blend, lowering the phase separation temperature, lowering the amount of ethanol which can overcome into the water phase, reducing the increase of vapor pressure behavior of gasoline due to the adverse influence of ethanol addition. [18] The phase separation results in significant gasoline quality degradation, since part of oxygen content is disappeared, octane value is reduced and volatility is changed causing problems at regular consumers.

The work of de Menezes E.W. et al [13] was the motive for the present investigation. The aim of the present work was to investigate the water tolerance of oxygenated ethanol-ETBE- gasoline blends. At first, ethanol was added in refinery fractions to determine their water tolerance. Consequently, these refinery fractions were suitably combined so as to simulate the composition of commercial gasoline. Ethanol was added into before-mentioned gasolines and the water saturation of oxygenated blends was examined. Then, ETBE was added as a co-solvent and the influence of ETBE addition on the water saturation of ethanol – gasoline blends was examined thoroughly. The results showed that the "in common" addition of ethanol and ETBE (up to 20% v/v) increased the water tolerance for the all gasoline compositions.

II. Materials And Methods

Three refinery fractions were supplied by Aspropirgos Refinery of Hellenic Petroleum SA. The refinery components examined, were:

Reformate: This component is the product of heavy naphtha catalytic reforming. Refomate contains mainly C7 to C10 hydrocarbons, has high content of aromatic hydrocarbons and has excellent octane rating characteristics with high RON and MON values and low volatility.

Isomerate: This component is produced from the conversion of normal paraffins contained in light naphtha to isoparaffin, containing mainly C5 and C6 isoparaffins. Isomerate is a component with low octane numbers and high volatility.

Table 1. Main Properties of the Refinery Blending Components

Property	Units	Reformate	Isomerate	FCC	Method
Density at 15 °C	kg/m^3	827.9	651.5	744.0	EN ISO 12185
Vapor Pressure at 37.8 °C	kPa	35.8	92.6	24.9	EN ISO 13016-1
RON		102.2	85.3	92.6	EN ISO 5164
MON		91.7	82.6	82.2	EN ISO 5163
Aromatics	% v/v	76.95	0.32	21.09	EN ISO 22854
Benzene	% v/v	0.73	0.13	0.77	EN ISO 22854
Olefins	% v/v			25.43	EN ISO 22854
n Parafins	% v/v	3.93	8.02	15.49	EN ISO 22854
iso Parafins	% v/v	17.22	82.38	23.23	EN ISO 22854
Naphthenes	% v/v	0.68	9.28	14.70	EN ISO 22854
Distillation					
IBP	°C	43.2	33.8	68.0	EN ISO 3405
10%	°C	84.8	42.0	74.2	EN ISO 3405
50%	°C	128.9	54.1	93.1	EN ISO 3405
90%	°C	162.2	112.1	139.9	EN ISO 3405
FBP	°C	191.1	157.0	159.3	EN ISO 3405
E70	% v/v	6.4	79.5	3.5	EN ISO 3405
E100	% v/v	19.2	88.0	59.6	EN ISO 3405
E150	% v/v	79.6	95.0	95.0	EN ISO 3405

Catalytic Cracking Naphtha (FCC): This process converts the heavy hydrocarbons of vacuum gasoil (VGO) into lighter and more valuable products, in the range of gasoline (C5 – C10 hydrocarbons) and some middle distillate components. Other side products are gaseous olefinic hydrocarbons (mainly C3 and C4). This is a fuel with very low volatility. The FCC Naphtha used in this series of experiments is the heavy naphtha that consists mainly of C6 to C10 hydrocarbons.

Table 2. Main Properties of the Gasoline Blends

Property	Units	G1	G2	G3	Method
Reformate	% v/v	40	50	30	
Isomerate	% v/v	20	20	20	
FCC	% v/v	40	30	50	
Density at 15 °C	kg/m^3	759.1	767.5	750.7	EN ISO 12185
Vapor Pressure at 37,8 °C	kPa	44.4	45.4	43.4	EN ISO 13016-1
RON		94.1	95.1	92.8	EN ISO 5164
MON		84.1	84.9	83.0	EN ISO 5163
Aromatics	% v/v`	39.28	44.87	33.69	EN ISO 22854
Benzene	% v/v	0.63	0.62	0.63	EN ISO 22854
Olefins	% v/v	10.17	7.63	12.72	EN ISO 22854
n Parafins	% v/v	9.37	8.22	10.53	EN ISO 22854
iso Parafins	% v/v	32.66	32.06	33.26	EN ISO 22854
Naphthenes	% v/v	8.01	6.61	9.41	EN ISO 22854
Distillation					
IBP	°C	45.9	47.8	52.1	EN ISO 3405
10%	°C	64.8	63.1	68.0	EN ISO 3405
50%	°C	105.9	110.3	103.3	EN ISO 3405
90%	°C	154.2	158.8	153.9	EN ISO 3405

FBP	°C	180.3	185.7	171.4	EN ISO 3405
E70	% v/v	14.8	14.4	12.5	EN ISO 3405
E100	% v/v	44.7	42.3	47.4	EN ISO 3405
E150	% v/v	87.1	84.9	87.5	EN ISO 3405

The physicochemical properties of three refinery fractions were presented in the Table 1. Apart from the refinery samples, fuel grade bioethanol that meets EN 15376, with purity 99.8% v/v, supplied by Tarımsal Kimya Teknolojileri A.Ş. (TARKİM), Turkey was used in this series of experiments. As fuel grade, the bioethanol sample used was "dry" with very low water content, and it was denaturated with ETBE. ETBE was supplied by Eni Ecofuel SpA, Italy.

The procedure followed for the preparation of oxygenated fuel blends was the following: Ethanol – Refinery fractions and ethanol – ETBE – gasoline were mixed in a glass beaker. Then, they were covered with aluminum foil and stirred in a magnetic stirrer on a plate. Water saturation tests were made by titration of water from a burette into the blends. The delivery of the volume of water to the system was followed by shaking to hasten the solubility. The water saturation point was judged to be reached when fine droplets were observed that could not be dissolved. All the experiments were conducted under thermostatic conditions at 20°C. The main properties of the gasoline blends formed are displayed in Table 2.

III. Results And Discussion

Ethanol was added in three refinery fractions such as Reformate, FCC Naphtha, Isomerate in order to be determined how much water these fractions tolerate. Ethanol was added up to 20% v/v using a step of 5%. The water retention of these refinery fractions is displayed in Fig. 1. The higher water tolerance was displayed by Reformate refinery fraction. The more polar hydrocarbons such as Reformate, due to the presence of π bonds in the molecule, form bonds with the polar ethanol molecules, increasing the miscibility, and as a result, the water tolerance of the blends. [15, 19]

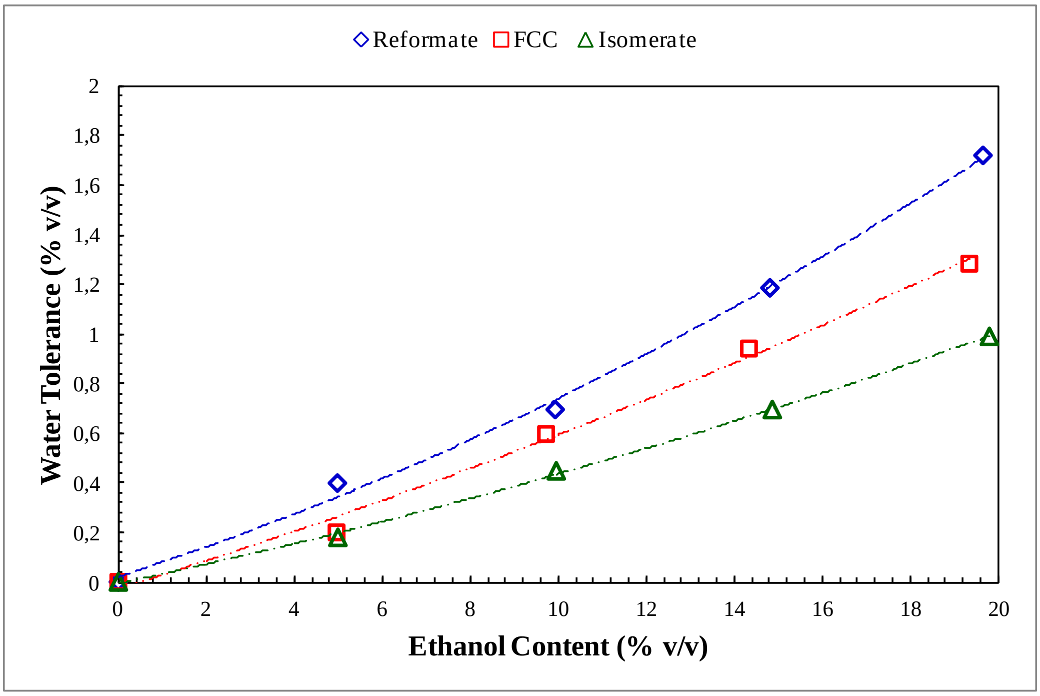

Figure 1: Water tolerance of refinery fractions

The FCC naphtha followed in water tolerance. FCC naphtha includes mainly C6 – C10 olefinic hydrocarbons which have a polar nature. The aromatic hydrocarbons content is significantly lower than that of the Reformate. This lower aromatics content seems to affect water retention, as the FCC naphtha seems to have worse affinity with ethanol, and the result is that this component retains less water when ethanol is added. [19, 20] The lower water retention was displayed by Isomerate fraction. This fraction contains mainly saturated hydrocarbons (and mainly isoparaffins) with near zero percentage of polar compounds, therefore, as it was expected, it retains very limited amount of water, even at high concentrations of ethanol. [16]

Consequently, a further step of investigation includes the preparation of commercial like gasoline samples by the combination of the three refinery fractions. These fractions were mixed in particular percentages.

In an attempt to examine the influence of gasoline composition on water tolerance, three gasoline samples were prepared with the following compositions: G1: 40% Reformate – 40% FCC Naphtha – 20% Isomerate, G2: 50% Reformate – 30% FCC Naphtha – 20% Isomerate, G3: 30% Reformate – 50% FCC Naphtha – 20% Isomerate. These concentrations were particularly designated in order to form gasoline samples with characteristics approaching those of commercial gasoline. The sum of polar containing compounds reached at 80% v/v, including FCC Naphtha and Reformate fractions and a smaller percentage of 20% v/v belonged to Isomerate, a non-polar compound which remained fixed in all the three blends that were prepared. The addition of ethanol in the three gasoline blends was carried out up to 20% v/v using a step of 2.5%. As recorded in published works, when the quantity of ethanol in gasoline increased the mixture had a greater tolerance for water and was more resistant to phase separation. [19]

The water retention of three gasoline blends is presented in Fig. 2. Subsequently, in these oxygenated blends ethanol- gasoline, ETBE was added up to 20% v/v.

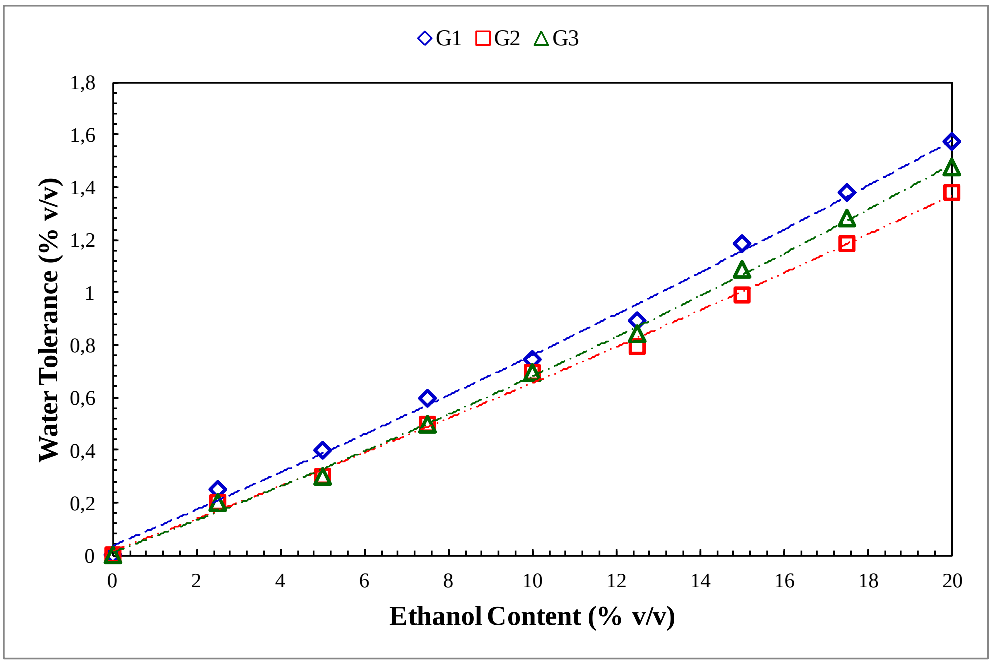

Figure 2: Water tolerance of G1, G2, G3 gasolines

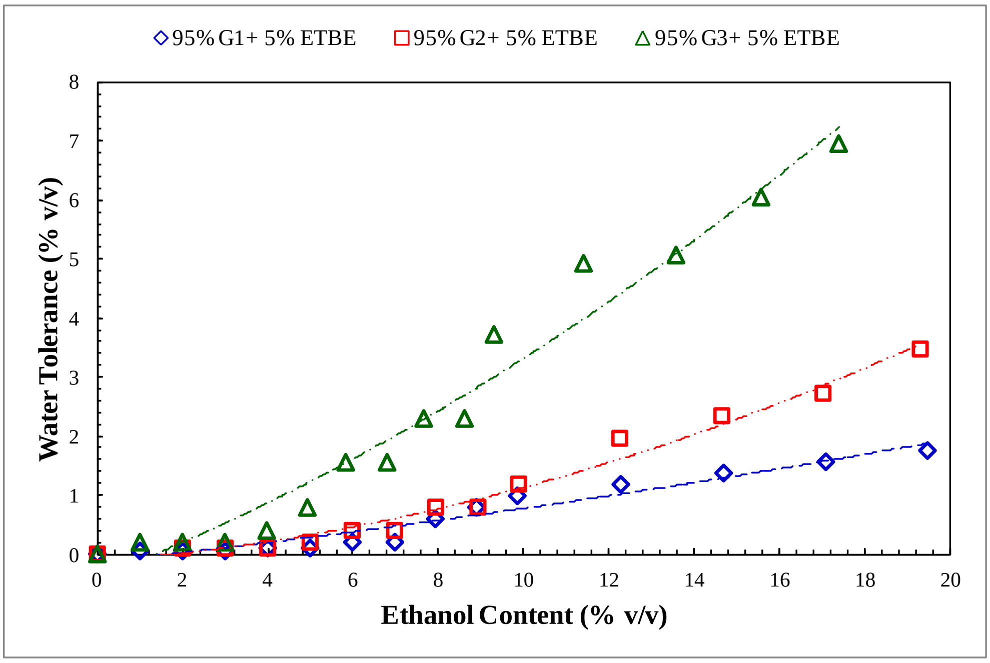

Figure 3: Water tolerance of gasolines with 5%v/v ETBE addition

The addition of ethanol in gasoline blends resulted in an increase of water retention for all the samples. The maximum water tolerance was recorded for the higher percentage of ethanol addition (20% v/v). Also, G1 gasoline showed a higher value of water tolerance, while G2, G3 displayed close behavior. The difference of water tolerance between gasolines was recorded for higher percentage of ethanol addition (>15% v/v). It is noted that the difference in the composition of the three blends does not seem to affect significantly the water tolerance, as small differences were observed. The gasoline blend with the higher water tolerance was G1 with equal amounts of Reformate and FCC Naphtha in the fuel composition (Fig. 2). Fuel blend G3 with the higher Reformate (and therefore aromatic hydrocarbons content) presented the lower water tolerance Fig. 2.

As mentioned before, ETBE was added in the base gasoline blends at different concentration, in an attempt to study the impact of ethanol and ETBE addition on water tolerance. The common presence of MTBE and ethanol has been beneficial in water tolerance, [21] therefore, it is expected that ETBE could have a similar behavior. This impact of ETBE addition on water tolerance of the three gasoline blends is displayed in Fig. 3, Fig. 4, and Fig. 5.

The maximum water tolerance for ETBE addition at 5% v/v was recorded at 20% v/v ethanol addition. G3 gasoline blend showed the maximum water retention at 6.96% v/v H_2O followed by G2 (3.47% v/v) and then G1 (1.75% v/v). The ETBE addition at 5% v/v interacted better with the fuel with higher olefins content (G3 blend) containing 50% FCC Naphtha and 30% Reformate, increasing the water retention of oxygenated blends as shown in Fig. 3.

Similar behavior was recorded for the addition of 10% v/v ETBE. For G3 gasoline blend, a higher value for water tolerance was noticed reaching 7.41% v/v H_2O followed by G1 (4.21% v/v) and then G2 (2.11% v/v). In the case of ETBE addition at 10%, the blend with the higher olefins content presented the higher water retention (Fig. 4).

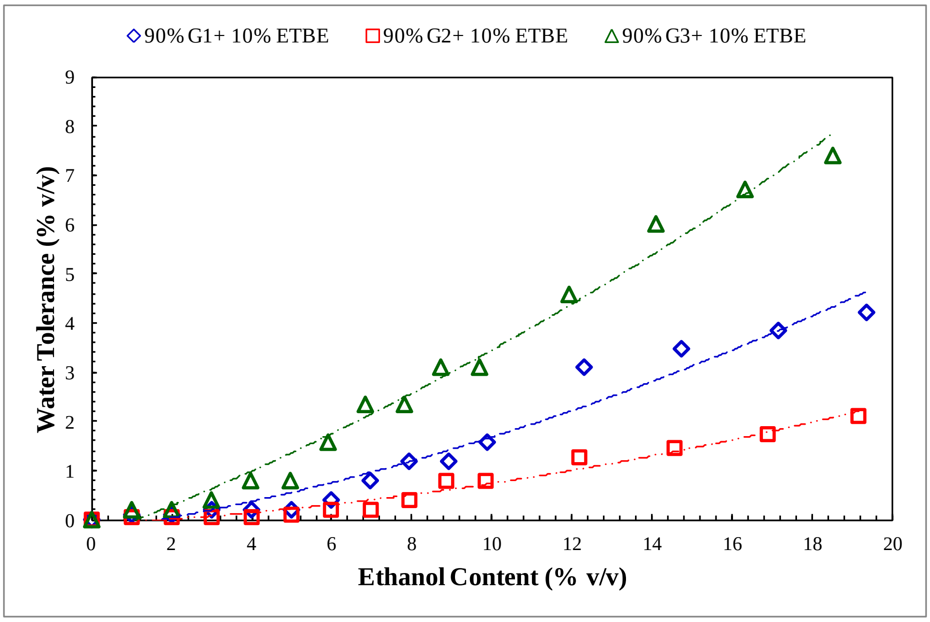

Figure 4: Water tolerance of gasolines with 10%v/v ETBE addition

In the case of a higher ETBE addition at 20% v/v, the three gasoline samples showed similar water retention implying that in this high concentration of ETBE the composition of gasoline doesn't play a significant role. The 'in common' presence of 20% v/v ETBE and ethanol led to an intermediate value of water retention reaching 4.94% v/v for G2, G3 and 4.58% v/v for G1 respectively. In such case, similar values of water tolerance for gasolines were recorded. (Fig. 5) and a significant decrease of water tolerance was noticed for G3 gasoline compared to the relevant values for a lower ETBE addition at 5 and 10% v/v.

The common presence of ethanol and ETBE in the gasoline blends led to an increase of water tolerance as displayed in Fig. 3, Fig. 4, and Fig. 5. In bibliography, the role of ETBE addition was as a co-solvent to increase the water retention of gasoline blends. Particularly, in a high ETBE addition of 15% v/v, the ether groups probably build hydrogen bonds with water increasing the water solubility. Specifically, when the ETBE content increased from 4 to 15% v/v the amount of water needed for phase separation at constant temperature was also increased. The higher amount of ETBE provided an increasing availability to form hydrogen bonds with water molecules. [22] The evaluation of the present results showed that the higher water saturation was observed at 20% ethanol and 10% ETBE addition reaching 7,40% v/v H_2O (Fig. 4).

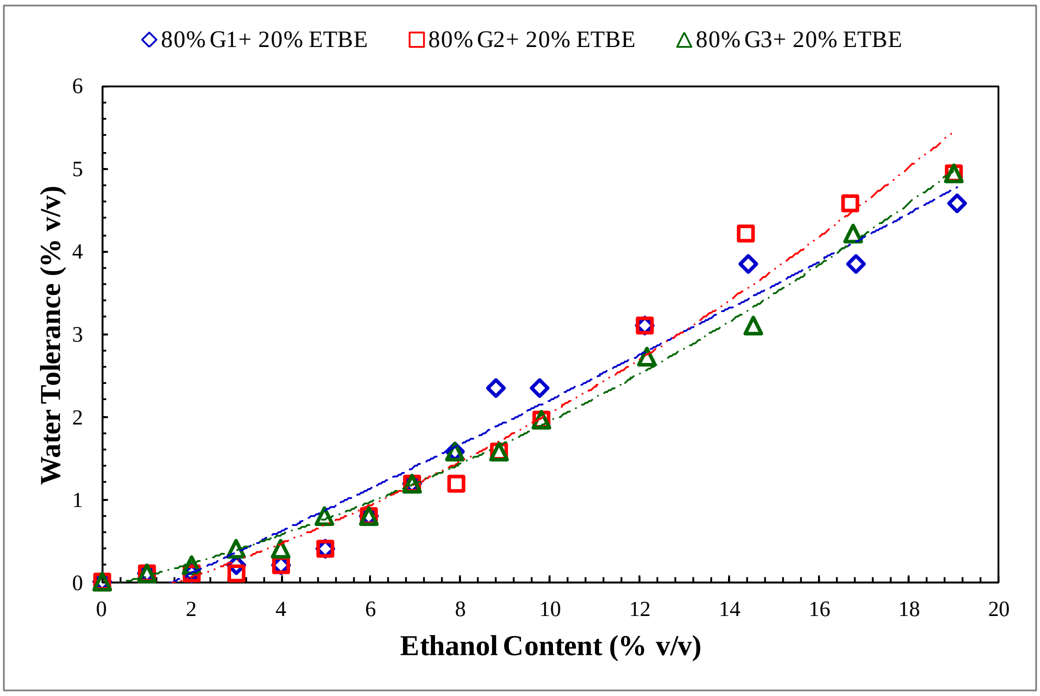

Figure 5: Water tolerance of gasolines with 20%v/v ETBE addition

It is worth noticing that in bibliography other co-solvents or combination of them were examined thoroughly. Tertiary-butanol in ethanol-gasoline blends led to an increase of water content. [23] Analogous behavior was faced with the combination of other co-solvents such as TAME and ETBE. Their presence in ethanol- gasoline blends promoted more tolerance to water. [24] The water solubility was increased slightly with the higher unsaturated hydrocarbons content and with the content of oxygenated co-solvent like ETBE. [14] Additionally, according to the published works, similar results were recorded in the addition of methanol with co-solvents in gasoline regarding the solubility of water in blends. As recorded, the solubility of water in methanol-gasoline blends, containing from 3% to 5% v/v methanol, is low. When TAME is added in the blend, the solubility of water increases. For comparison reasons, the solubilizing effect of two ethers, namely TAME and MTBE, on the solubility of water in blends containing 90% v/v gasoline, 5% v/v methanol and 5% v/v ether, did not show any difference between the ethers. [25] According to published works, the presence of MTBE and ETBE was beneficial, since they slightly increased the miscibility of 1-butanol gasoline blend with water and decreased the temperature of the phase separation. Ethanol acted in the same way increasing significantly the water solubility. [26]

In Fig. 6, Fig. 7, and Fig. 8 the influence of ethanol addition (0-20% v/v) and ETBE addition (0, 5, 10, 20 % v/v) is displayed on each gasoline blend individually. The G1, G2 gasoline samples showed the maximum water retention at 20% v/v ethanol addition and 20% v/v ETBE addition (G1: 4,58% and G2: 4.94%) approaching similar values. For G1 gasoline a close behavior was recorded for 10% v/v ETBE addition (4.21% v/v) and much lower values were noticed for 5% v/v ETBE addition (1,75% v/v) as depicted in Fig. 6. For G2 gasoline, the addition of ETBE showed a different behavior. The 5% v/v ETBE addition (3.47% v/v) followed 20% v/v ETBE addition and then 10% ETBE addition reaching 2.11% (Fig. 7). In case of G3 gasoline the maximum water tolerance was recorded at 20% v/v ethanol and 5% v/v or 10% v/v ETBE addition showing similar water tolerance at 6.96% and 7.41% v/v H_2O respectively. Lower values were noticed for 20% v/v ETBE addition reaching only 4.94% v/v water retention (Fig. 8).

As shown in Fig. 6, Fig. 7, and Fig. 8, there is a difference in the water tolerance of the three gasoline blends when ethanol and ETBE were added. As mentioned above in Table 2, the gasoline blends have quite different compositions. G1 and G2 samples have similar behavior (water tolerance is increased by increasing ETBE content), but for G3 sample, water tolerance is lower for the high ETBE addition (20% v/v). G3 sample has the lower aromatics content and the higher olefins content compared to the other two gasoline blends. Olefinic hydrocarbons have lower water tolerance compare to aromatic hydrocarbons, and this may be an explanation to this different behavior of this less aromatic gasoline blend. [20]

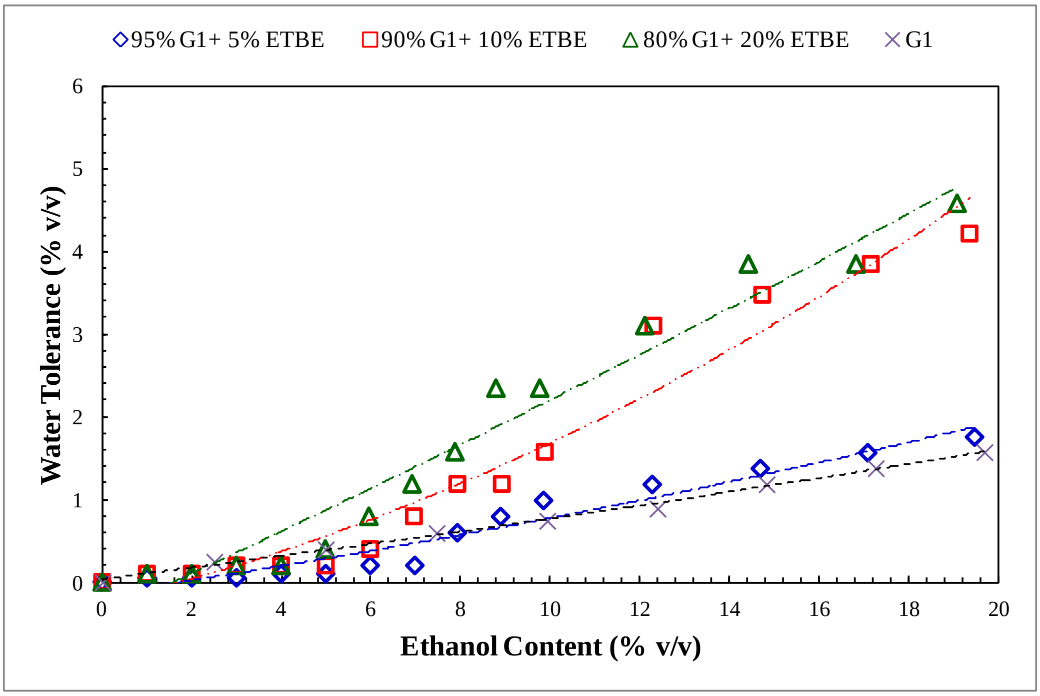

Figure 6: Water tolerance of G1 gasoline

In any case, it is worth commenting that the common presence of oxygenates led to a significant increase of water tolerance of gasoline samples. Particularly, the maximum increase of water retention due to the presence of oxygenates was noticed for G1: from 1.57 to 4.58% v/v at 20% ethanol and 20% ETBE addition, for G2: from 1.38% to 4.94% v/v at 20% ethanol and 20% ETBE addition, for G3: from 1.48% to 7.41% v/v at 20% ethanol and 10% ETBE addition. (Fig. 6, Fig. 7, Fig. 8)

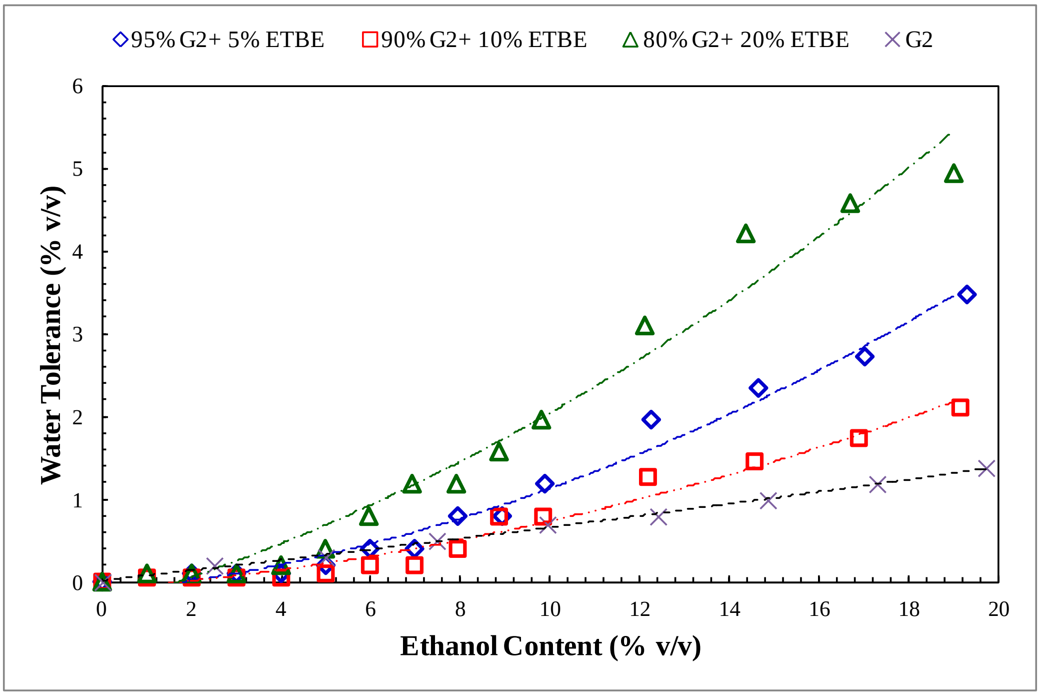

Figure 7: Water tolerance of G2 gasoline

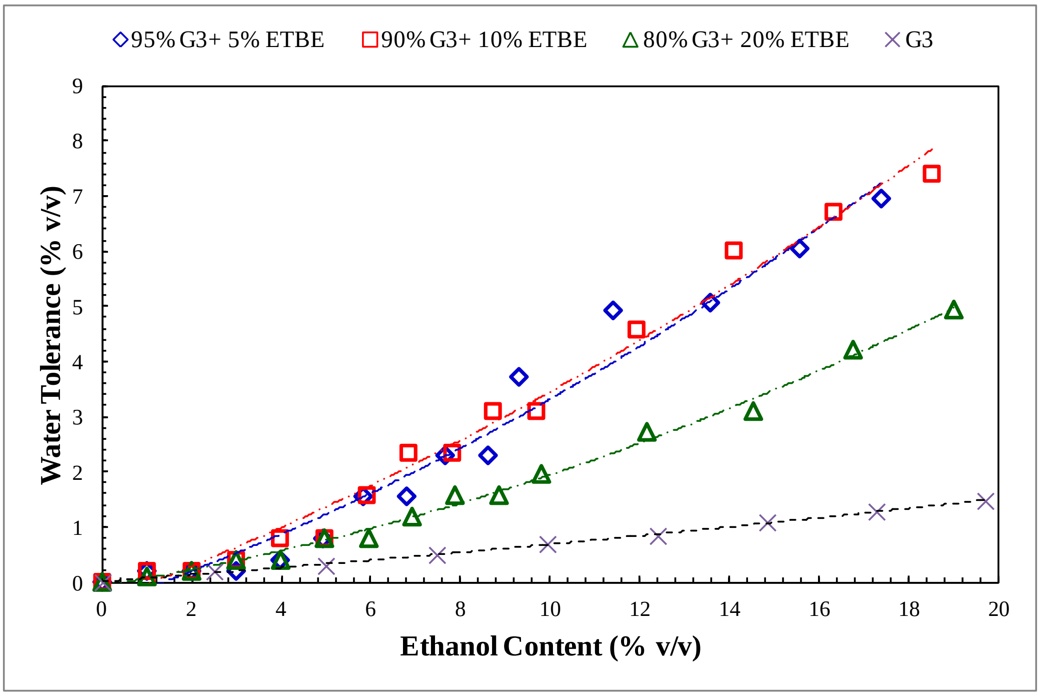

Figure 8: Water tolerance of G3 gasoline

IV. Conclusion

In the present work the water tolerance of ethanol-ETBE-gasoline blends was investigated. In the ethanol-gasoline blends, ETBE was added as a co-solvent and the aim of the present work was to investigate the influence of ETBE addition as a co-solvent on the water saturation of ethanol-gasoline blends.

Gasolines containing ethanol have a high affinity to water, due to the high polarity of ethanol. The aim of the present work was to increase the water tolerance by combing ETBE as co-solvent in order to avoid any phase separation. This separation may result in a significant decrease of ethanol content in fuel blend and loses of gasoline quality via a decrease of octane number value.

From the results of experiments of this specific work, it was recorded that the water tolerance was affected by the composition of the gasoline. The results showed that the addition of ethanol on polar compounds such as Reformate and FCC resulted in a higher tendency to keep water in a stable dissolved phase compared to the non- polar compounds (like normal and iso-parafins), which they have the lowest water retention compared to the other types of hydrocarbons.

ETBE is recommended as a co-solvent to enhance the overall behavior of the gasoline so that ethanol and water to be drawn into the hydrocarbon phase improving their solubility, without phase separation. The present results showed that the higher water retention was recorded for the common presence of ethanol and ETBE in the gasoline. For two of the three gasoline samples that were tested, the better water tolerance was achieved for the blends with high content in both ethanol and ETBE. The third sample, with lower aromatics content and higher olefins content presented the better water tolerance for the lower ETBE contents (5% and 10% v/v).

Summarizing, it was found that the common presence of ETBE with ethanol in gasoline can be helpful not only in octane number and volatility of the blends, but also in improving the water retention. This improvement can be very important for the quality of the fuel that is distributed to the end consumer.

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