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Chapter

Structural Integrity of Materials in Fuel Ethanol Environments

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Abstract

Nowadays, the use of liquid fuels is prevalent in the transport sector due to ease of storage. There are two different fuel types namely, fuels obtained from fossil resources and biofuels made from renewable resources. Typical biofuels in industry use include pure plant oil (PPO), biodiesel, ethyl tertiary butyl ether (ETBE), biobutanol and fuel ethanol. Studies carried out have shown that fuel ethanol can substitute petrol. In addition, ethanol can be blended with gasoline at any ratio depending on the circumstances and the desired fuel. Typical fuel ethanol blends in use are: E5, E10, E20, E25, E70, E85, E95 and E100. Remarkably, there have been evidences of stress corrosion cracking (SCC) of steel storage tanks and associated piping used in fuel ethanol service during the past decade. This chapter is therefore, centered on a description of structural integrity issues related to metallic and non-metallic materials in fuel ethanol environments. Prior research on the corrosion and stress corrosion cracking behavior of ethanol-gasoline blends are also reviewed.

Keywords: fuel ethanol, materials, structural integrity, fracture, corrosion

1. Introduction

There is often a great deal of corrosion data on a number of engineered materials. However, much of the available data is clustered in a limited number of environments, full immersion environments in particular. The report of the National Research Council in the United States (US) [1] revealed that the limited number of environments for corrosion research has resulted in inability to create a meaningful national database of corrosion data useful to industry, government and academia. Aside from the issue of full immersion, atmospheric and alternate immersion aqueous environments, there are also completely different environments such as non-aqueous and high-temperature environments. Ethanol is an example of non-aqueous environments for which a better ability to predict its influence on various engineering materials is paramount due to its planned widespread use.

One of the key drivers for the development of biofuels globally is the concern about universal climate change, which is mainly instigated by combustion of fossil fuels. Considerable scientific evidence abounds indicating greenhouse gas (GHG) emissions as the reason for accelerating global warming. Biofuels are not only renewable and viable energy sources but are toxic-free and so more environmentally friendly than conventional petroleum-based fuels [2, 3]. Biofuels are also biodegradable and therefore their inadvertent spillage is of no significant environmental hazard [2–4]. While biodiesel and PPO are appropriate for diesel engines,

Fuel	Density	Viscosity	Flashpoint	Calorie value at 20°C MJ/kg	Calorie value MJ/l	Octane number RON	Fuel equivalence
	kg/L	mm ² /s	°C		MJ/l		1
Petrol	0.76	0.6	<21	42.7	32.45	92	1
Fuel ethanol	0.79	1.5	<21	26.8	21.17	>100	0.65

Table 1.
Parameters of fuel ethanol in comparison with petrol [6].

fuel ethanol can replace petrol [5–7]. The properties of fuel ethanol are shown in **Table 1** and compared with the properties of fossil petrol.

The anti-knocking property of the fuel is influenced by the octane number while its energy yield is about one third lower than petrol. Ethanol, also known as ethyl alcohol ($\text{CH}_3\text{CH}_2\text{OH}$) is a volatile, flammable, colorless liquid obtained from some energy crop that comprises high quantities of sugar or substance that can be converted into sugar like starch or cellulose from grains [6]. In the US the most common source is from corn and grain. In Brazil, it is sourced from sugarcane [8].

However, ethanol can also be produced naturally (fermented) from any carbohydrate source, such as wheat, cane, beet and fruits like grapes and apples [8]. While grain and synthetic alcohols are technically the same (the molecule is identical), there are differences in the amounts of contaminants (butanol, acetone, methanol, organic acids) in each. According to Paul and Kemnitz [9], for ethanol to be used as fuel, water must be removed. If fuel ethanol is vended with zero water content, it would be referred to as anhydrous ethanol. Typically, denatured alcohol holds about 1% water besides additional constituents. Fuel ethanol with <0.5% water is considered “anhydrous ethanol” [8]. Ethanol with higher water content is usually referred to as “hydrated ethanol”. Such hydrated ethanol is uncommon in the US but has been used as a fuel in Brazil.

During the past 8 years, a substantial testing effort on the structural integrity of metallic and non-metallic materials in fuel ethanol has been undertaken by various organizations. Though SCC has not been extensive, it has caused several failures in a number of user facilities. Various factors have been associated with ethanol SCC of carbon steels which include: conditions that promote crack initiation and growth, dissolved oxygen concentration levels, chloride concentration, corrosion potential, water content, and the chemical species of the ethanol itself.

There have been a substantial number of notched slow-strain rate (N-SSR) tests conducted with the aim of studying stress corrosion crack initiation (SCCI) and propagation mechanisms in fuel ethanol [10]. It is worth noting that significant concerns currently exist regarding the SCC behavior of pipeline steels as well as terminal facilities used to handle fuel ethanol.

2. Stress corrosion cracking in fuel ethanol environments

A corrosion failure such as stress corrosion cracking is an insidious form of corrosion which has far more adverse effects. Usually there is no prior warning before failure due to SCC. A 2004 survey of causes for failure in refining and petrochemical plants in Japan shows that a majority of the failures were due to corrosion, with the highest percentage due to SCC [11, 12]. The chart in **Figure 1** shows percentages of failures by type of material of construction [11]. Stress-corrosion failures can affect public health as in pollution due to escaping product from corroded

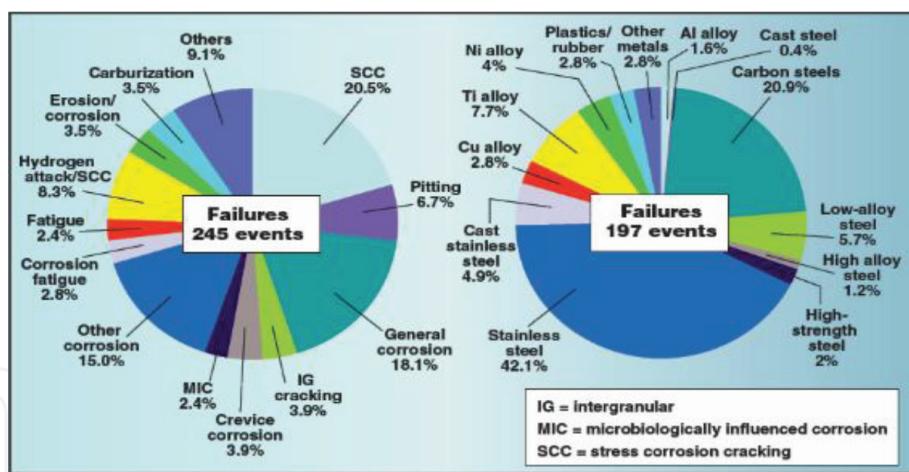


Figure 1.
Component failure frequencies [12].

equipment or due to the corrosion product itself. Sudden failure could result into fire, explosion, release of toxic products and construction collapse [1, 12, 13].

Commencing just about 2002, a number of ethanol storage tanks at blending terminals which have been used for a period of <2 years suffered leaks owing to SCC [14, 15]. Afterwards, more than 35 incidences of SCC failures in tanks, associated piping, and fittings have been discovered by an industry survey [14]. All failures so far have been in blending terminals, occurring in several regions in the United States. No SCC case has been reported by ethanol producers, transportation trucks, service stations and rail cars. Brazil has manufactured and distributed ethanol for quite a few years and has not likewise reported any SCC. Because of these failures, there was concern about the ability of pipelines to safely transport ethanol to and from blending terminals.

2.1 Supply chain of fuel ethanol

As soon as fuel ethanol is produced at a manufacturer's facility, it is held in storage tanks pending its release for distribution. Generally, manufacturers add the denaturant before or in the course of onsite storage. In addition, an inhibitor is added during storage or just preceding discharge of the shipment for supply. This may be one reason for SCC experience at some downstream facilities and no reported failures at manufacturer facilities. On entering the distribution system, fuel ethanol can be transported by numerous means, which include pipeline, barge, tanker truck and railroad tanker car [16].

The duration that fuel ethanol spends in the sequence can fluctuate significantly from days to months, subject to several factors: the obtainability of intermediate distribution storage, the site of the manufacturing facility, the transportation mode used, and the location of gasoline blending terminals. Fuel ethanol is held in storage tanks as soon as it comes into a gasoline blending facility. Contingent on usage and traffic requirements, the residence period in these tanks also differs. In certain cases, it can be held for months in the course of a period of dormancy [16].

However, in certain instances, at gasoline blending facilities, the residence period in the storage tank is relatively short as incoming ethanol supplies and outgoing shipments of blended gasoline are a proximate frequent process. Nevertheless, observations of SCC have been restricted to the lot of the supply chain encompassing the intermediate liquids storage through the gasoline blending facility and possibly will be linked to circumstances that develop in the distribution system or variations that transpire in the fuel ethanol [16].

2.2 Documented cases of SCC in fuel ethanol

Research carried out by the American Petroleum Institute (API) has shown that SCC of steel in fuel ethanol environment is a subject matter where awareness of the issue is growing dynamically as a result of documentation of experiences and research works in progress. Findings by API point out that documented catastrophes of ethanol process equipment dates back to no less than the early 1990s. Establishments undergoing what they contemplate as cases of SCC in fuel ethanol have been stimulated to confirm these issues through appraisal and documentation of service conditions, along with metallurgical examination of the failed or cracked components.

The appearance of cracks caused by other cracking environments is similar to SCC cracks of steel in fuel ethanol. Instances of SCC in steel equipment exposed to fuel ethanol are presented in **Figures 2–4**. The cracks are characteristically branched and may possibly be transgranular, intergranular or mixed mode.

Both transgranular and intergranular cracking may well occur in laboratory testing subject to the composition of ethanol. However, greater number of cracks documented from field failures display intergranular cracking. While analyzing a field catastrophe, intergranular cracking suggests ethanol SCC, but transgranular or mixed mode cracking might likewise be present [16].

Instances of SCC of steel components in fuel ethanol have been conveyed in the following kinds of equipment in gasoline blending facilities and fuel ethanol distribution:

- a. Welds and adjacent metal in tank bottoms, detached roofs besides related seal components;
- b. Fittings, facility rack piping, and accompanying equipment (for example, air eliminators);
- c. Nozzle welds and vertical seam in lower tank shells situated off bottom;
- d. Pipeline used to convey fuel ethanol from terminal to end user facility.

The blend of low cost and strength brands carbon steel as the principal material of construction for equipment used in the conveyance, handling and storage of fuel ethanol [16]. Generally, carbon steel is thought as compatible with fuel ethanol from the perspective of corrosion since its corrosion rates are characteristically low. On the other hand, the corrosion rate can occasionally escalate with agitation, the presence of contaminants, and the level of dissolved oxygen content of the ethanol. In the API program, the field corrosion rate measurements in fuel ethanol point out that the corrosion rates of carbon steel were typically very low.

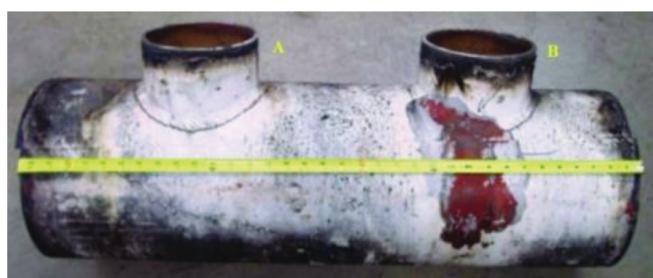


Figure 2.

Locations of ethanol SCC near fillet welds used to make the branch connections to piping B [17].



Figure 3.
Photograph of cracked steel elbow welded to the flange [17].

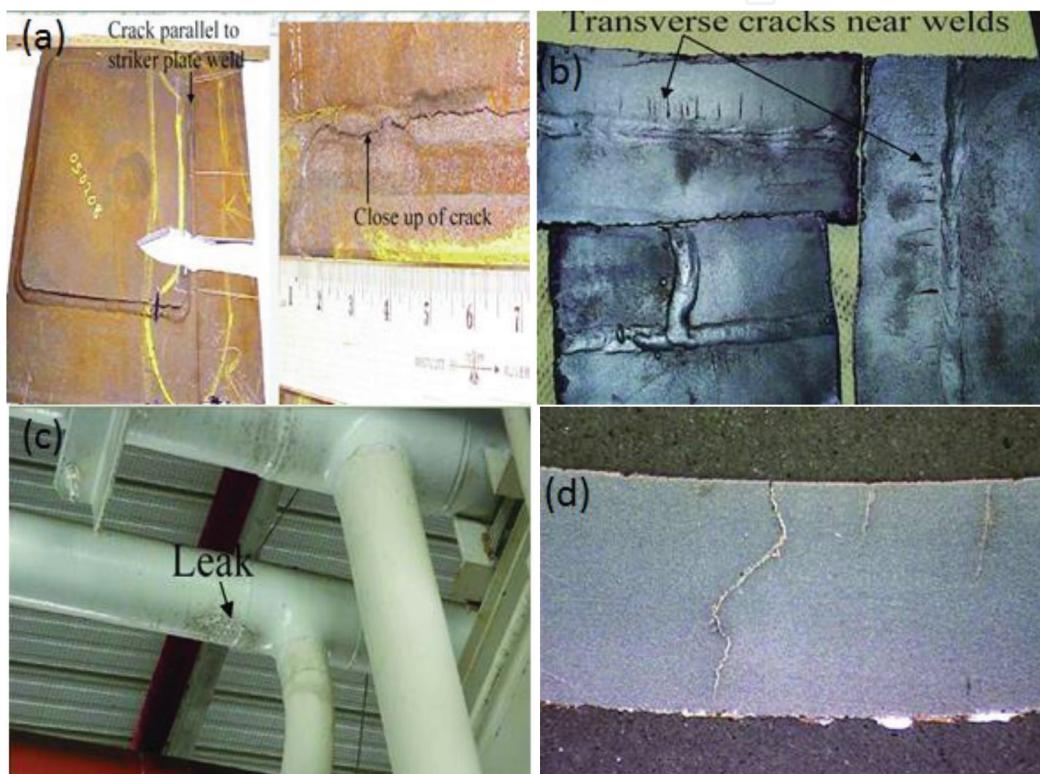


Figure 4.
SCC failures showing (a) SCC in steel tank bottom, (b) SCC in steel air eliminator vessel, (c) leak in piping resulting from a crack adjacent to the weld, (d) multiple crack initiations and through-thickness propagation in piping [8, 16].

2.3 Structural integrity of materials in fuel ethanol environments, previous research and current trends

Investigation of the corrosion and stress corrosion cracking (SCC) mechanism of steel in fuel ethanol is still in the early stages and several countries are considering increasing biofuel production as an approach to secure future energy supplies and mitigate global warming. When these come to the market, the infrastructure will play a key role in ensuring safe, reliable, and efficient distribution of these fuels to the end users [14]. Pipeline is the most effective transportation method in meeting these requirements. Hence, there is dire need of evaluating and predicting the influence of fuel ethanol on various steel grades which can be used for such pipelines.

A most recent study [18], jointly funded by API and Renewable Fuels Association (RFA), using the slow strain rate test method (SSRT), found that SCC of steel can take place in fuel ethanol meeting the ASTM D4806 standard

specification (see **Table 2**). From the study, the inhibitor, Octel DC1-11 was discovered to lower the corrosion rate of steel in ethanol but had no effect on SCC. In addition, the team found that in addition to water, the most important factor that caused SCC in fuel ethanol appeared to be dissolved oxygen. When dissolved oxygen was minimized through nitrogen purging, no SCC occurred in the presence of all other species at their maximum levels. But on introducing oxygen, the reverse occurred. Furthermore, corrosion potential was used to monitor the potential for SCC of steel exposed to ethanol. One short coming of the study was that the results obtained are limited to fuel ethanol of ASTM D4806 standard and the study of the effect of stress level on SCC was left out. Hence, parameters for estimating risk of SCC from known defects in the studied environment were not obtained.

Other studies include those of Beavers et al. [19] and Lou et al. [20]. While [19] examined pitting corrosion in simulated fuel grade ethanol (SFGE) solutions on carbon steel, [20] examined the addition of chemical additives to SFGE to provide scavenging of oxygen in solution or inhibition of SCC in fuel grade ethanol (FGE) using slow strain rate (SSR) techniques. The latter study found a dependence of ethanol SCC on electrochemical potential that was consistent with observations from previous API studies (i.e., increased susceptibility to SCC with increasing corrosion potential). Based on this study, three active techniques of non-chemical deaeration were recognized. Altogether, the three methods reduced the corrosion potential below -100 mV Ag/AgCl EtOH and alleviated SCC.

Also, Beavers and Gui [21] summarized the results of research studies involving factors affecting ethanol SCC of carbon steel as water content, level of aeration, aging during storage, blend ratio with gasoline, steel type and welding. In addition, Gui et al. [22] carried out studies on the influence of ethanol composition on SCC susceptibility of carbon steel by evaluating ethanol SCC in field FGE samples and correlating the results in terms of SCC severity to compositional differences in the FGE samples. Carbon steel was found to be susceptible in all FGE samples conducted in two laboratories but with a varied degree of susceptibility in one FGE sample compared with the others.

Furthermore, Venkatesh et al. [10] evaluated the SCC behavior of pipeline steel in multiple ethanol environments. The program used N-SSR testing and field samples of FGE obtained from Brazilian sources. Severity of cracking was assessed

Property	Units	Specification	ASTM designation
Ethanol	%v min	92.1	D5501
Methanol	%v max	0.5	—
Solvent-washed gum	mg/100 ml max	5	D381
Water content	%v max	1	E203
Denaturant content	%v min, %v max	1.96, 5.00	D4806
Inorganic chloride	ppm (mg/l) max	40 (32)	E512
Copper content	Mg/kg max	0.1	D1688
Acidity as acetic acid	%m (mg/l)	0.007 (56)	D1613
pH	—	6.5–9.0	D6423
Appearance	Visibly free of suspended or precipitated contaminants (e.g., clear and bright)		

Table 2.
Quality specifications of fuel ethanol per ASTM D4806 [16].

based on crack growth rates determined from N-SSR testing and K_{ISCC} values based on a fracture mechanics treatment of the N-SSR test data. In another study [23], the effects of inorganic chloride in ethanolic solutions on the SCC behavior of carbon steels was assessed by varying the inorganic chloride concentrations between 0 and 70 mg/L using additions of sodium chloride (NaCl) to SFGE. The results indicated that both crack density and crack growth rate increased with chloride concentration. Two laboratory testing programs were used to evaluate the SCC behavior of steel in fuel ethanol and butanol [24]. The first part of the program revealed that cracking of API 5L X42 carbon steel compact tension specimens in FGE solutions (client supplied and synthetically prepared) required high K (stress intensity) values to initiate cracks. Highest crack growth rates were observed in SSR tests and in tests conducted in SFGE and under aerated conditions. Fracture mechanics tests and tests involving an actual field sample of FGE resulted in lower crack growth rates.

The second part of the program evaluated ASTM A36 carbon steel for SCC in the reagent grade butanol and anhydrous butanol solutions using SSR testing. The tests showed no evidence of SCC. Likewise, Cao [25] studied the corrosion and stress corrosion cracking of carbon steel in simulated fuel grade ethanol using SSR techniques and accurately controlled fracture mechanics conditions. Goodman and Singh [26] evaluated the influences of chemical composition of ethanol fuel on carbon steel pipelines using SSR testing on carbon steel samples in five FGE environments. SCC was discovered in two of the as-received FGE environments and in FGE environments to which NaCl was added.

Furthermore, substantial information has been gathered from reviews, reports and summaries of studies investigating the compatibility of fuel ethanol with metallic materials. Nevertheless, care must be taken in interpretation of the information [27]. Examples are:

- a. a Concawe [28] report recommending carbon steel and aluminum for ethanol/petrol handling situations; and
- b. a laboratory study conducted by Minnesota Pollution Control Agency [29] evaluated 19 metallic species, including four types of aluminum alloy and brass in E10 and E20 blends, three aluminum alloys were adjudged as satisfactory as was brass.

Unfortunately, it is known from field experience that E10 blends can severely corrode aluminum components, leading to catastrophic failure [27, 30]. Also, carbon steel can suffer severe corrosive attack if the fuel contains water [27, 31]. Likewise, brass components in carburetors are known to corrode when exposed to E10. The carburetor manufacturer who reported this, conducted compatibility testing of its products with petrol/ethanol blends and has identified corrosion of metallic components as an issue, requiring replacement of brass components with more resistant, but more expensive, alloys.

Qinetiq reports the Brazilian experience with ethanol blends [27, 32]. According to Stephen [27], in order to make vehicles more durable when employing ethanol blends, various fuel system components require modifications among which are:

- a. zinc steel alloy fuel lines changed to cadmium brass;
- b. tin and lead coatings (terne plate) of fuel tanks changed to pure tin; and
- c. cast iron valve housings changed to iron cobalt alloy (QINETIQ, 2010).

Beavers et al. [33] carried out a recent research that was funded by the Pipeline Research Council, in which methods for prevention of internal SCC in ethanol pipelines were evaluated. The methods assessed include the addition of inhibitors and oxygen scavengers to ethanol and other ways and means of deaeration. On the other hand, Beavers et al. [34] studied the effects of ethanol-gasoline blends, metallurgical variables, inhibitors and dissolved oxygen on the stress-corrosion cracking of carbon steel in ethanol. Slow strain rate (SSR) and fatigue precracked compact tension (CT) tests were employed to characterize the influence of environmental and metallurgical variables on SCC of carbon steel. Metallurgical factors, including steel grade within a range of pipeline grades, welds, and heat-affected zone, do not seem to have a noteworthy effect on the degree or frequency of SCC. In terms of environmental factors, it was observed that SCC of carbon steel does not take place even in a completely aerated state, if the ethanol-gasoline blends contain below approximately 15 vol.% ethanol; susceptibility to SCC and crack growth rate are greater in 50 vol.% ethanol gasoline blend (E-50) than in either lower or higher ethanol concentration blends; oxygen scavenging can be an effective method to inhibit SCC; water content exceeding 4.5 wt.% prevents SCC in ethanol; and fatigue precracked CT tests display comparable inclinations to SCC susceptibility as SSR tests.

Maldonado and Kane [35] studied the stress corrosion cracking of carbon steel in fuel ethanol service and postulated that the hygroscopic nature of ethanol is an important aspect with potential relevance to its corrosivity. Also, ethanol possesses high potential for oxygen solubility; therefore, the availability of oxygen for involvement in the corrosion reaction is anticipated to be largely greater.

The authors in [36] presented an evaluation of fatigue crack propagation in three steels namely; A36, X52 and X70 steels in a SFGE. By using a fracture mechanics approach to determine crack propagation rates, all the three materials were found to be prone to enhanced fatigue damage in fuel-grade ethanol environments.

Figure 5 shows a macroscopic view of the fracture surface of X52 steel after testing in SFGE. A model for determining crack growth rates in ethanol fuel was further proposed by the authors.

A recent study [37] investigated the corrosion of martensitic stainless steel in ethanol-containing gasoline mixture as a function of water, chloride and acetic acid concentrations. The results obtained showed that, water and chloride ions (Cl^-) are the primary corrosion causing factors in EtOH/gasoline mixtures; critical water

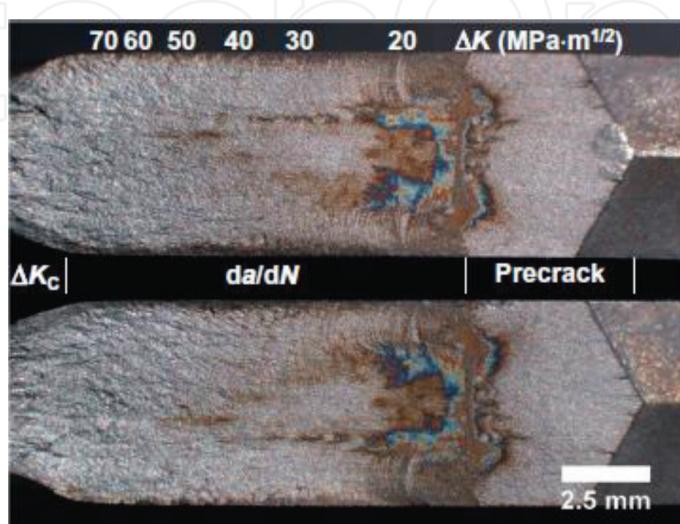


Figure 5.
Macroscopic view of X52 fracture surface after testing in SFGE [36].

content depends on EtOH/gasoline-ratio; pitting corrosion occurred at tremendously low chloride concentrations; increasing chloride concentration enhanced pit propagation, with slight influence on pit densities and higher concentrations of acetic acid lead to a greater attacked area, with negligible impact on the depth of pit propagation.

Another study [38] investigated the influence and role of minor constituents (organic acids, water and chloride) of fuel grade ethanol on corrosion behavior of carbon steel using X-ray photoelectron spectroscopy (XPS), auger electron spectroscopy (AES) and electrochemical experiments. The results showed that iron (II) acetate is generated on oxide film due to its high solubility in FGE environments. Chloride stimulated anodic dissolution at those sites where iron (II) acetate occurred.

Also, in 2016, Rangel et al. [39] carried out a study on the SCC susceptibility of API X-80 pipeline steel in SFGE. Water contents of 0, 1, 5, 10 and 20 vol.% and chloride content of 0, 10 and 32 g/L were investigated. Results have shown that X-80 carbon steel in the as-received condition was susceptible only when 5% water and 10 g/L NaCl were present. Heat treatments suppressed this susceptibility. Conditions that increased the corrosion rate also increased the SCC susceptibility, which, together with metallographic observations and noise in current measurements, indicated that SCC in this environment is caused by a film rupture, dissolution mechanism.

Recently, an investigation on the fracture behavior of micro-alloyed steel and API-5L X65 steel in simulated fuel ethanol environment was carried out [40]. Micro-alloyed steel was found to exhibit better fracture resistance than API-5L X65 steel in air and in solution. API-5L X65 in solution showed faster crack extension than MAS-in solution. It was also observed that J_{str} (fracture toughness derived from stretch zone geometry) obtained for the two steels shows a similar trend with J_i (initiation fracture toughness) which is found at the parting of the blunting line on their J-R curves and as a result appropriate for signifying the initiation toughness of the two steels in solution. On the whole, fuel ethanol decreases fracture resistance in X65 and micro-alloyed steels (**Figure 6**).

All of the findings point to the fact that SCC of metals do occur in FGE environment, whether simulated or field FGE due to several factors which have been mentioned. Most of the SCC tests were carried out using SSR techniques to assess the fracture toughness of the materials in fuel ethanol environment.

Ethanol fuels have gradually developed into a remarkable alternate energy source. Ethanol-based biofuel can be used to power engines and run cars, hence it is now the main alternative to automotive fossil fuels. The combination of gasoline

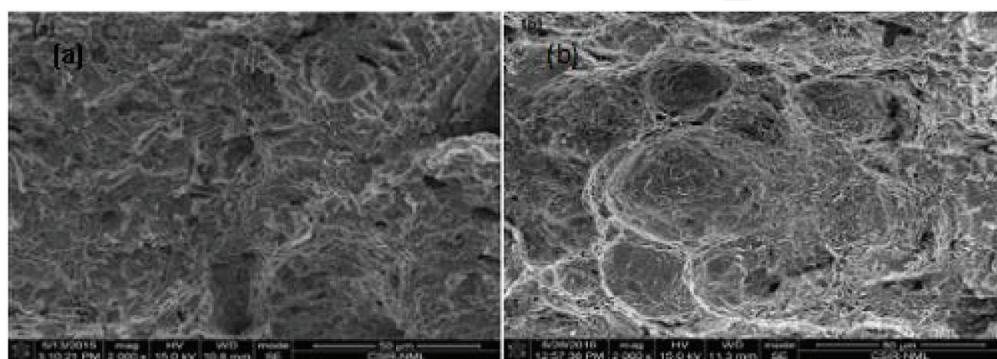


Figure 6.
Fracture surface of micro-alloyed steel and API-5L X65 steel after J tests in E20 SFGE [40].

with ethanol results into the fuel currently called “**Gasohol**” [4]. Despite the documented cases of corrosion and stress-corrosion failures in fuel ethanol, corrosion rates are typically low and recommendations regarding compatible materials are currently in literature [7, 40, 41]. These materials include carbon steels; micro-alloyed steel; unplated steel; stainless steel; black iron; bronze; polypropylene; Teflon; neoprene rubber; thermoplastic piping; thermoset reinforced fiberglass; nitrile and viton among many others. Hence, ethanol fuel is still the best possible alternative to fossil fuels.

Most of the gasoline sold in the United States contain some percentages of ethanol.

3. Conclusions

The kinetics of corrosion behavior, fracture behavior and crack growth depends on the material-environment system. It is important to state that function, material, shape and process do interact. The specification of process limits the materials you can use and the shapes they can take. In other words, the process of employing fuel ethanol in the fuel industry and its associated corrosion and stress corrosion failures has invariably placed a limit on the materials that can be used as pipes, storage tanks and the required automotive parts.

The structural integrity assessments carried out in fuel ethanol is of optimal benefit to designers in the fuel, automotive, aviation, and chemical industries. Material compatibility with fuel ethanol, based on corrosion rates, stress intensity factor, fracture toughness and crack propagation resistance, amongst others have been reviewed. A designer must give considerable attention to these parameters in order to ensure reliable performance of materials.

Requirements for design, materials and inspection are then established in a conventional manner relative to the estimates of progressive crack extension behavior presented in literature.

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Conflict of interest

The author declares no conflict of interest.

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