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Evaluation of Inulin and Aloe Vera as Green Corrosion Inhibitors for Mild Steel in 15% HCl

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Abstract

REACH legislation and PARCOM recommendations are driving research in environmental friendly alternatives to the highly toxic compounds currently used as corrosion inhibitors. Here two candidate plant extract green corrosion inhibitors are evaluated by direct comparison with commercially used corrosion inhibitors propargyl alcohol and 2-mercaptobenzimidazole.
The two candidate green corrosion inhibitors are: 1. commercially available powdered inulin, extracted from Jerusalem artichoke; 2. aloe vera gel extracted directly from the plants.
Immersion tests and weight loss measurements are used to determine the behaviour and inhibition efficiency as a function of concentration and temperatures of 20 ^{0}C to 60 ^{0}C for mild steel in 15% HCl.
Results show that inulin and aloe vera act as corrosion inhibitors over the range of conditions used, the optimal concentration for both was 10%, compared to 0.4% for the commercial corrosion inhibitors. Inhibition efficiencies of up to 86% and 84% were observed for inulin and aloe vera respectively, compared to values of over 95% for the commercial corrosion inhibitors. There is some variation of behaviour for inulin and aloe vera with time and temperature.

Keywords
Corrosion inhibition, sustainability, green corrosion inhibitors

Introduction

Mild steel is a common engineering material that finds application in several industries due to its excellent mechanical properties (good tensile strength, malleability, ductility), availability and relatively low cost[1]. However, it is severely corroded when exposed to aggressive conditions such as acid pickling, acid cleaning, stimulation of oil and gas wells, acid storage and de-scaling industrial processes, owing to its poor resistance to corrosion [2]. Cathodic protection, material selection, coatings, proper design and the use of inhibitors have all been employed to prevent the corrosion of mild steel, with the use of corrosion inhibitors proving to be one of the most efficient, practical and economical methods [3]. The major pre-requisites for the selection of inhibitor compounds are the ability of the inhibitor to oxidize the metal, the presence of functional groups containing hetero-atoms such as N, S, O and P which can donate their lone pair electrons, also the presence of π-bond which can provide the required electrons that will interact with the empty d-orbitals of the metal, the ability to cover a large metal surface area with a firmly attached protective film, inhibitors' cost effectiveness, solubility of the inhibitor and, increasing, environmental friendliness [4].
Synthesized organic compounds are the most widely used corrosion inhibitors due to their high inhibition efficiency in aggressive environment, however their replacement becomes necessary because they do not satisfy environmental and safety regulations on toxicity, bio-degradability and bio-accumulation [5]. Interest in the use of natural products such as plant extracts as green corrosion inhibitors, is increasing because of their easy availability, cost effectiveness, simple extraction procedures, biodegradability and environmental friendliness [5-8]. Plant extracts containing phytochemicals such as alkaloids, phenols, tannin, flavonoids etc. have been identified as of particular potential for use as green corrosion inhibitors. Such candidate corrosion inhibitors include:

Phoenix dactylifera and lactuca [9], pineapple leaves [10], dodonaea viscosa [11], jatropha curcas [12], nypa fruticans wurmb[13], date palm seed [5], nicotiana tabacum [14], murraya koenigii leaves [15], justicia gendarussa [16], ginkgo leaves [17] and aloe vera [18].
Aloe vera has been reported to act as a corrosion inhibitor for copper in 2M HCl and 2M H_2SO_4 [18], for zinc in 2 M HCl and on mild steel in 1 M HCl [19]. Hart and James[18] observed that the inhibition efficiency increased with concentration of the aloe vera gel: at 60^0C, the maximum inhibition efficiency of approximately 50% was seen when 10vol% of the gel was used. The corrosion inhibition effect of aloe vera gel has been attributed to the many oxygen atoms in its organic compounds such as aloin, aloesin, and aloeresin with functional groups O-H, C=O, and C-O [30]. The potential of inulin as a corrosion inhibitor was highlighted in work on chicory, inulin being the main constituent of chicory root [20]. Chicory was seen to be an effective corrosion inhibitor in 15% HCl at temperatures up to 93°C [20]. The molecular structure of inulin is shown in Fig. 1 below [23], the many hydroxyl (OH) functional groups in the molecule make it an attractive candidate corrosion inhibitor.

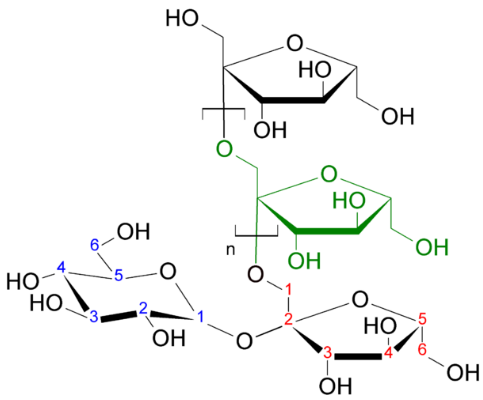

Figure 1: *Inulin molecular structure* ***[23]***

Some variation of the effectiveness of green corrosion inhibitors with temperature has been previously reported. For zenthoxylum alatum plant extract in 15% HCl inhibition efficiency decreased from 85 to 45% when the temperature increases from 30 to 80^0C [8]. The effectiveness of corrosion inhibition of gelatin (water-soluble polymer) on mild steel in 1 M HCl was noted to reduce as temperature increased due to disbonding of the polypeptide chain from the metal surface. Increasing temperature from 30°C to 60°C decreased the effectiveness of aqueous extract of morinda tinctoria on corrosion inhibition for copper in 0.5 M HCl, from 71% to 63% [21]. The effectiveness of aloe vera gel has also been reported to decrease as temperature increases [18, 19] [22]. For 10% aloe vera gel in 2 M HCl inhibition efficiency dropped from 67 to 60% when the temperature increased from 30^0C to 40^0C [22].
Variation with time has also been reported. For morinda tinctoria extract inhibition efficiency increased from 70. % at 24h to 81 % at 96 h [21]. The inhibition efficiency of aloe vera has also been reported to increase with time, and then plateau [19].
In this present study, the optimal concentrations and effect of temperature and time on the corrosion inhibition of two green plant based corrosion inhibitors: inulin (IN), and aloe vera (AV), on mild steel in 15%wt HCl solution are investigated, and compared with the commercially available synthetic corrosion inhibitors, propargyl alcohol (PA) and 2-mercaptobenzimidazole (2-MI). In this work the inulin used was obtained commercially as a powder whereas aloe vera was extracted directly from the plant.

Material preparation

As received mild steel rectangular coupons of size 6.0 cm x 4.0 cm x 0.3 cm were used for the weight loss measurements. The coupons were degreased in acetone, washed thoroughly with distilled water and air dried. The edges and bottom of the mild steel coupons were coated with lacquer and kept in a desiccator. Samples were used within 24h of preparation. Chemical compositional analysis of the mild steel specimen was determined by spark emission spectrometry the results presented in Table 1 are averages over seven separate measurements.

Table 1 *Spark emission chemical composition results of as received mild steel.*

Element	C	Mn	S	P	Pb	Mo	Al	Cr	S	W	Fe
Weight %	0.03	0.17	0.01	0.01	0.05	0.01	0.02	0.01	0.01	0.08	99.6

Acid solution preparation

The test solution of 15 % w/w HCl was prepared by diluting analytical reagent grade 37 % HCl (ThermoFisher Scientific) with double distilled water.

Inhibitors

Propargyl alcohol (99% purity) and 2-mercaptobenzimidazole (98% purity) were procured from Sigma Aldrich. Inulin powder (90% inulin, gelatin and rice flour) was obtained in powder form from Swanson Health Products. Aloe vera gel was extracted directly from aloe vera plants. As shown in fig. 2 standard aloe vera pot plants were used, with individual leaves ranging from approximately 10 to 25cm in length. Extraction was done simply by cutting the leaves open with a knife and squeezing the gel out. 320ml of gel was extracted from 676.5g of fresh aloe vera leaves, this was the total obtained from two plants. The aloe vera gel was used within 30 minutes of extraction.

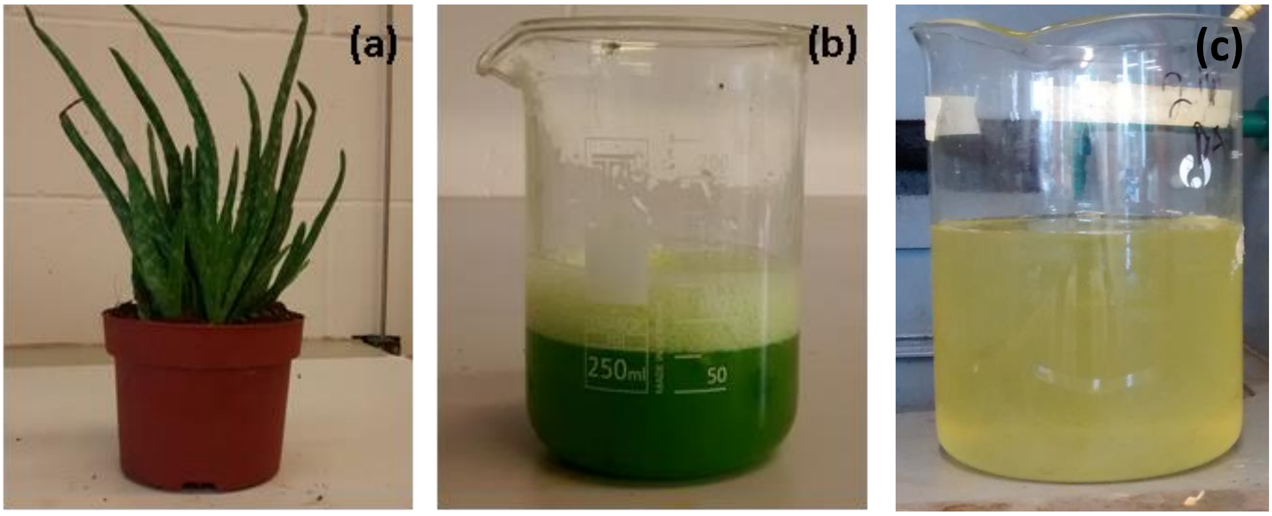

Figure 2: *(a) Aloe vera plant; (b) extracted gel; (c) 15% w/w HCl with 10% aloe vera gel.*

Test solutions were prepared by mixing appropriate quantities of the various inhibitors with 15% HCl. Concentrations of 0.1 to 10% were used.

Weight loss measurements

Three samples were used for each test. These were fully immersed in 3000ml of the test solution. Tests were run using inhibitor concentrations of 0.1 - 10% for times of 5 to 24 at temperatures of 20 to 60°C. Tests were also run in the absence of any corrosion inhibitor. A water bath was used to fix the temperature of the solution. The average value of the weight loss was used to calculate the corrosion rate (CR) and inhibition efficiency (η) using the following equations[24, 25]:

$$\text{CR (mm/y)} = \frac{(87600 \times \Delta W)}{(A \times T \times D)} \quad \textbf{(1)}$$

Where, ΔW = weight loss (g), A = area of specimen exposed (cm^2), T = time of immersion (h), and D = density of mild steel (g / cm^3), 87600 is simply a constant to convert the result into mm/y.

$$\eta\ (\%) = \frac{C_a - C_i}{C_a} \times 100 \quad \textbf{(2)}$$

Where, Ca and C_i are the corrosion rates of mild steel in the absence and presence of inhibitor, respectively.

Results and Discussion

Effect of concentration on corrosion inhibition of mild steel

The variation of inhibition efficiency as a function of inhibitor concentration for samples immersed at 20°C for 5 h are shown in Fig. 3. As expected [27, 28], the inhibition efficiency increased for all inhibitors as the concentration used increased, until a plateau was reached. This can simply be attributed to an increase extent of adsorption of inhibitor species at the metal-solution interface [29]. It is seen that the different corrosion inhibitors have different optimal concentrations, i.e. different concentrations beyond which there is no further improvement in inhibition efficiency for the given conditions. For propargyl alcohol and 2-mercaptobenzimidazole this optimal concentration was seen to be 0.4%, a value in line with the literature [31]. It is clear that considerably higher concentrations are required for both candidate green corrosion inhibitors. Both inulin and aloe vera have optimal concentrations of 10%. The optimal concentrations found here were used throughout the rest of this work.

Table 2 shows corrosion rates for the mild steel coupons, extrapolated from 5 hour tests at 20°C. Whilst the inhibitors (inulin and aloe vera gel) reduced the corrosion rate by nearly an order of magnitude, these values are still much higher than those considered desirable for the oil and gas pipeline industry However, the corrosion rate is more significantly by the propargyl alcohol and 2-mercaptobenzimidazole: the corrosion rate of mild steel is 0.1 mm/y for 0.7% propargyl alcohol, which is in line with the values desired in the oil and gas industry where inhibited corrosion rates of 0.1 mm/y are quoted in a recent standard [33], though other articles quote corrosion rates of around 3-5 mm/y [34]. Obviously actual corrosion rates are heavily dependent on test conditions and the corrosion rates in Table 2 are perhaps best regarded a comparative measure.

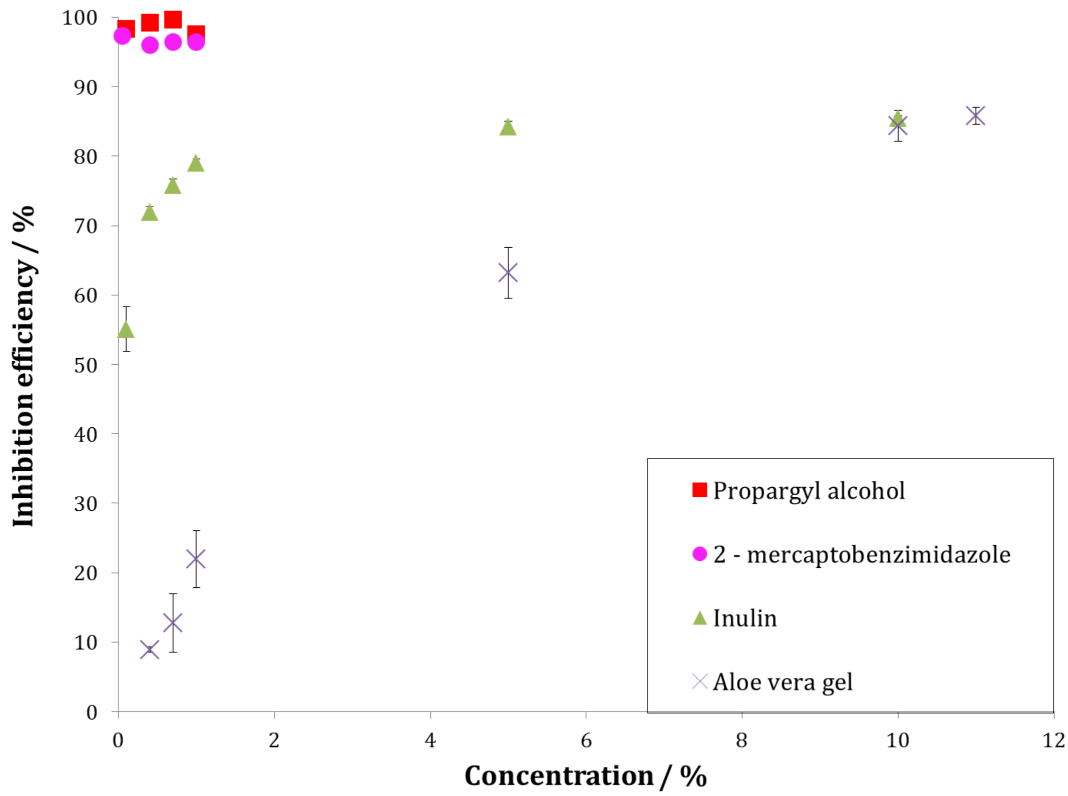

Figure 3: *Variation of inhibition efficiency with concentrations of PA, 2-MI, IN and AV at 20^{0}C for 5h. Error bars show standard deviation.*

Table 2 *Corrosion parameters of mild steel immersed in 15% w/w HCl solution in the absence and presence of different concentrations of inhibitors at 20^{0}C for 5 h.*

Inhibitor	**Concentration (%)**	**Weight loss (g)**	**Corrosion rate (mm/y)**
Control	0.0	0.2601	30.11
Propargyl alcohol	0.1	0.0045	0.52
	0.4	0.0020	0.23
	0.7	0.0009	0.10
	1.0	0.0022	0.20
2- mercaptobenzimidazole	0.1	0.0165	1.91
	0.4	0.0105	1.21
	0.7	0.0091	1.05
	1.0	0.0094	1.09
Inulin	0.4	0.0729	8.44
	0.7	0.0630	7.29
	1.0	0.0530	6.14
	5.0	0.0430	4.72
	10.0	0.0377	4.36
Aloe vera gel	0.4	0.2369	27.43
	0.7	0.2268	26.26
	1.0	0.2028	23.48
	5.0	0.0957	11.08
	10.0	0.0405	4.69

Effect of time of immersion on the corrosion inhibition of mild steel

The effect of variation of inhibition efficiency with immersion time for the various corrosion inhibitors at 20°C is shown in Fig. 4. No change in inhibition efficiency was seen for either propargyl alcohol or 2-mercaptobenzimidazole, this is consistent with the literature [36] and is due to strong adsorption of the inhibitors on the surface of mild steel forming a stable protective film [35]. There is a suggestion of some drop off in inhibition efficiency for both inulin and aloe vera over the 24 h of the test.

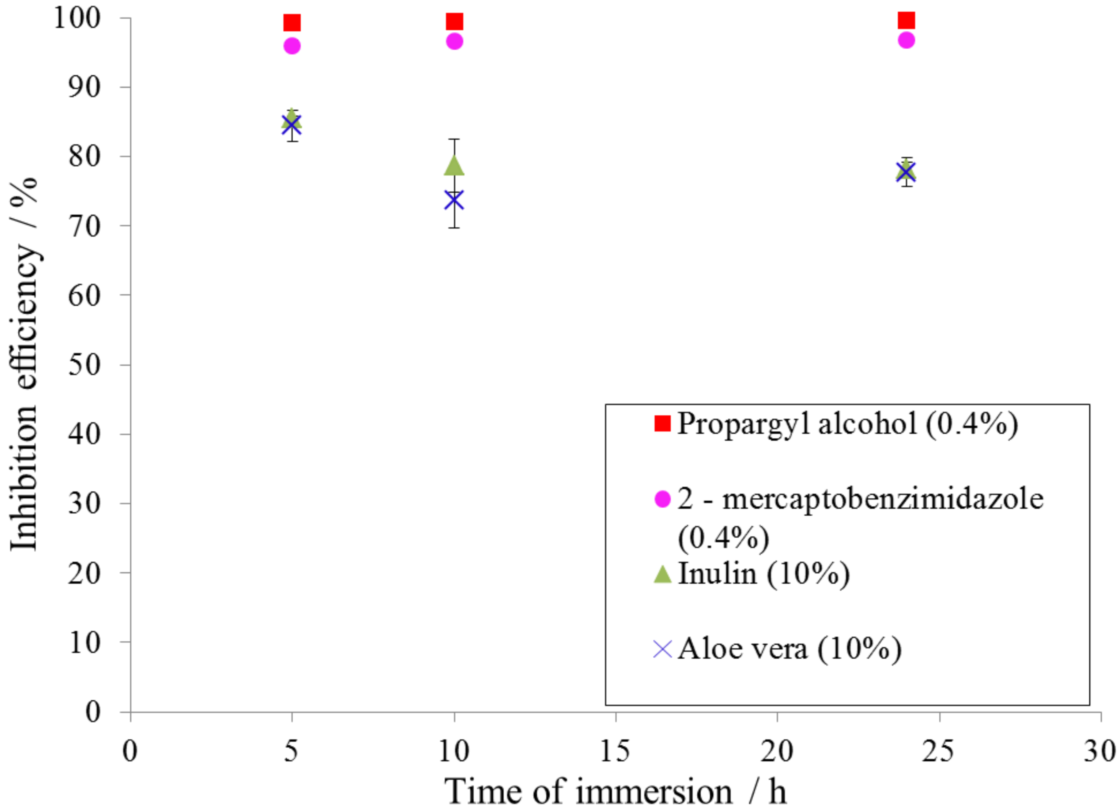

Figure 4: *Variation of inhibition efficiency with immersion time of PA, 2-MI, IN and AV on mild steel in 15% w/w HCl at 20^{0}C.*

Effect of temperature on the corrosion inhibition performance

The effect of temperature on the effectiveness of the corrosion inhibitors is shown in Fig. 5. Propargyl alcohol shows no significant variation in behavior over the temperature range used. The corrosion inhibition performance of 2-mercaptobenzimidazole decreased as the temperature increases, with the inhibition efficiency dropping from 96% at 20°C to 88.4% at 60°C. A similar trend has previously been reported [38] and is probably due to increased thermally activated desorption of the inhibitor molecules from the metal surface with increasing temperature.

The two green corrosion inhibitors behave differently: the inhibition efficiency of inulin increased with temperature, whereas that of aloe vera decreased between 20°C and 40°C with little further change as temperature increased to 60°C.

It must be noted that only data for 5 h immersion times are shown and were shown in Fig. 4 there is some variation with time for both inulin and aloe vera. Further work getting results from different time points at the higher temperatures is required to get a full understanding of the performance of these candidate green corrosion inhibitors.

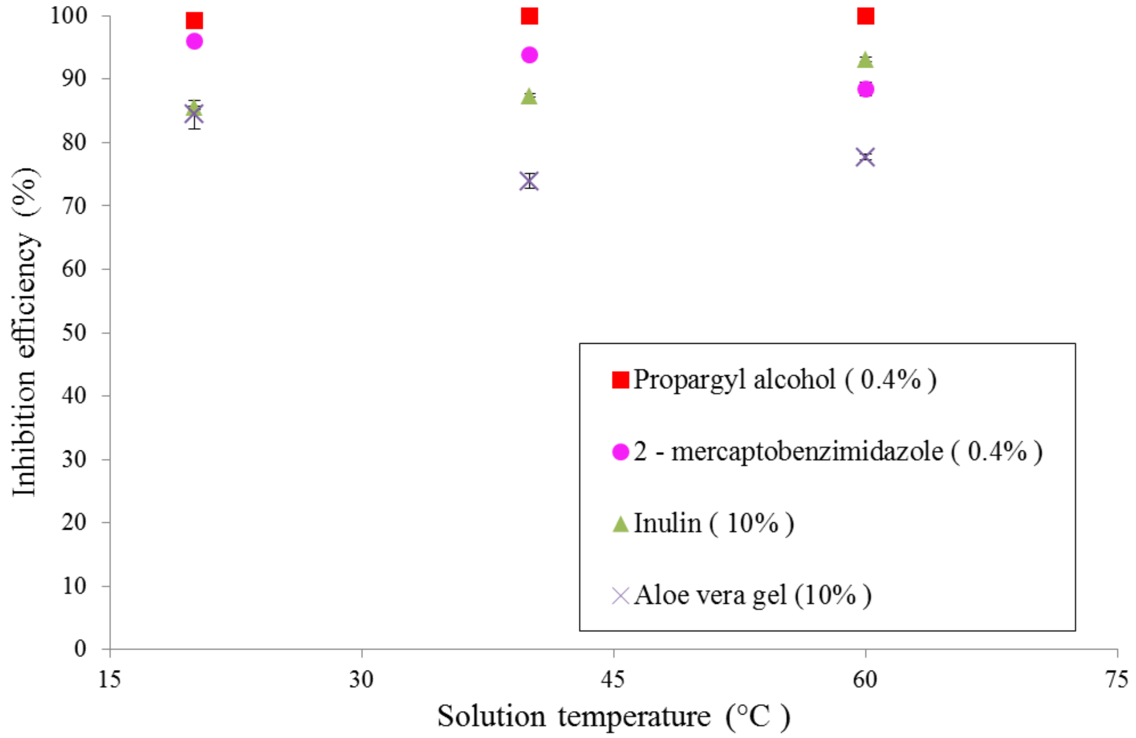

Figure 5: *Variation of inhibition efficiency with solution temperature at optimum concentration of PA, 2- MI, IN and AV for 5h.*

Discussion

The results presented in this paper show that both inulin and aloe vera can act as corrosion inhibitors for the conditions used. However both must be used in significantly higher concentrations than currently commercially available corrosion inhibitors. The inhibition efficiencies are also typically 10-15% less than those of the commercially available corrosion inhibitors. It is clear that aloe vera and inulin are not yet able to compete with commercially available corrosion inhibitors for the conditions used. Further investigation and development is required.

This work has shown some effect of time and temperature on the performance of the green corrosion inhibitors. While this is mainly attributed to attachment and detachment at the metal surface there is some concern about degradation of the aloe vera gel. Further work will study the variation of performance with time and temperature as well as time since extraction and the potential for addition of suitable preservatives. A different aspect of aloe vera is whether or not any further processing of the gel could enhance its performance, by changing the dispersion of active species or by breaking down cells etc.

Some consideration of the commercial viability of using these green corrosion inhibitors is required. The simplicity of the aloe vera extraction process and the ready availability of powdered inulin are positive points. Aloe vera has numerous applications so extraction of the gel is already done on a commercial scale. This is advantageous, however the demand for aloe vera for competing applications could be a problem as it will increase the price. Price is always going to be a key factor, particularly given the increased quantities of inulin or aloe vera that appear to be required compared to current commercially available corrosion inhibitors. Further work will also include characterization of the absorbed film, covering the effect of time and temperature. This will enable inulin and aloe vera to be optimized as corrosion inhibitors, something that is required before full commercial evaluation can be done.

A recent review noted that the inhibition action of many, if not all, the green inhibitors examined so far is "*purely dependent on the metal type and composition of solution*" [40] this dependency on the specific system means that further work on different systems is required for full investigation of both candidate green corrosion inhibitors.

Conclusions

- Inulin and aloe vera can act as corrosion inhibitors in 15% HCl over the temperature range of 20- 60°C.
- The optimal concentration for inulin and aloe vera was 10%, compared to 0.4% for the commercial corrosion inhibitors.
- Inulin and aloe vera may have a slight drop off in inhibition efficiency with time.
- The performance of inulin and aloe vera is influenced by changes in temperature over the temperature range of 20- 60°C.
- Further data is required for full understanding of how inulin and aloe vera perform as corrosion inhibitors.

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