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Performance evaluation of an imidazoline corrosion inhibitor in a CO₂-saturated environment with emphasis on localised corrosion

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Abstract

The ability of an imidazoline inhibitor to provide corrosion protection to X65 carbon steel in a CO₂-saturated sodium chloride (NaCl) solution at 80°C was evaluated. The performance of the inhibitor was investigated with respect to both generalised and localised corrosion behaviour using *in-situ* electrochemical measurements, *ex-situ* scanning electron microscopy (SEM) and non-contact surface profilometry. We demonstrate that the optimum inhibitor concentration required to minimise generalised corrosion is significantly lower than that required to minimise localised corrosion. This highlights the importance of systematic, post-test topographical measurements in the selection and optimisation of inhibitor dosage.

Key words: CO₂ corrosion, imidazoline corrosion inhibitor, pre-corrosion, localised corrosion

1.0 Introduction

It is essential to optimise the efficiency of various methods designed to minimise corrosion during hydrocarbon exploration, development and production operations in the oil and gas industry. One of the most common methods is the application of corrosion inhibitors which can protect typical carbon steels during multi-phase production operations. Such corrosion inhibitors are deployed via continuous injection systems, batch treatments or squeeze treatments. The majority of these corrosion inhibitors comprise nitrogen-containing compounds which function by forming a protective film on the steel surface. It is important to select an inhibitor formulation which provides both optimum performance under the prevailing corrosive conditions and compliance with various regulations associated with the location and environment of deployment.

Corrosion inhibitor formulations must be properly evaluated by thorough laboratory tests before application in the field. All such inhibitors should be tested for compatibility with the corrosive environment and should provide efficient protection against corrosion without any antagonistic effects with other chemicals in the process fluid. In terms of a proper evaluation of inhibitor performance, it is important to perform rigorous testing with respect to both generalised and localised corrosion behaviour in order to ensure both corrosion mechanisms are successfully mitigated [1, 2].

In general, organic corrosion inhibitors which exhibit film forming properties are surface active compounds comprising a hydrophilic polar head group at one end of the molecule and a nonpolar hydrophobic tail at the other. The polar head group adsorbs on the steel surface and the hydrophobic tail is oriented away from the surface. Close packing of the hydrophobic tails creates a hydrophobic surface [2], and the efficiency of these inhibitors is highly dependent on the extent of surface coverage by the protective film. Typically, there is a direct relationship between the inhibitor concentration or dosage and the efficiency of protection; the efficiency of protection increases with increasing inhibitor concentration until a maximum is achieved. However, this optimum value is highly dependent upon the operating conditions, particularly the operating temperature. For example, high temperature, biodegradable corrosion inhibitors effective at temperatures above 100°C were tested by Jenkins [3] with the aim of developing environmentally friendly corrosion inhibitors for a gas/condensate field project. These inhibitors were required to minimise corrosion in a subsea carbon steel pipeline under CO₂-saturated multiphase flow at temperatures above 100°C. Jenkins [3] tested the performance of three modified quaternary amine compounds and reported that higher inhibitor dosages were required at higher temperatures, e.g. 100 ppm was required at 120°C compared to 35 ppm at 100°C. The requirement for higher dosages was partially attributed to the formation of a FeCO₃ corrosion product on the steel surface at 120°C.

Most published studies report on the efficiency of inhibitors with respect to generalised corrosion with very few studies reporting on both generalised and localised corrosion behaviour [4]. Quantification of generalised corrosion behaviour provides essential information on inhibitor adsorption kinetics, efficiency and mechanisms but it is important to quantify the ability of the inhibitor to minimise localised corrosion. Ultimately, a high proportion of failures in the field are attributed to localised corrosion as opposed to a generalised decrease in pipeline wall thickness [5].

Guldbrandsen *et al.* [6] demonstrated the importance of quantifying localised corrosion of low alloy steels by evaluating the effect of pre-corrosion on the performance of two imidazoline and two amine-based corrosion inhibitors using potentiodynamic polarisation and electrochemical impedance measurements. Experiments were carried out in CO₂-saturated environments at 20 and 50°C in 1 and 3 wt.% NaCl solutions at pH 5. Experiments were performed under static conditions in a glass cell and in a glass loop with pre-corrosion periods of up to 18 days. In their

study they evaluated the performance of the aforementioned inhibitors on three different materials; X65 carbon steel, 0.5% Cr steel and St52 steel. Their results indicated that increasing either pre-corrosion time or temperature led to a decrease in inhibitor efficiency and localised corrosion in the form of deep pits on the steel surfaces, as observed by SEM. Guldbrandsen *et al.* [6] reported that the negative effect of pre-corrosion appeared to be related to the development of cementite protrusions on the steel surface and concluded that the decrease in inhibitor performance with increasing pre-corrosion time is co-determined by the properties of the steel and the composition of the corrosion inhibitor. Similar observations were made by Paolinelli *et al.* [7] and Zhang *et al.* [8] who reported that an increase in pre-corrosion time led to a decrease in inhibitor efficiency which was attributed to the formation of corrosion products. However Senatore *et al.* [9] observed a notable synergistic interaction between the inhibitor and the corrosion product in providing protection against erosion-corrosion.

Palmer *et al.* [10] employed an alternative approach to assess the ability of corrosion inhibitors to minimise localised corrosion. In their study, an artificial pit electrode was utilised to simulate a pit along with zero resistance ammetry (ZRA) and polarisation/depolarisation resistance measurements to determine the ability of the inhibitor to retard corrosion within the artificial pit. The experiments were conducted in hydrogen sulphide (H₂S) and CO₂ environments under simulated barium sulphate scaling conditions at ambient temperature and at 55°C, respectively. Palmer *et al.* [10] reported that once localised corrosion (pitting) had been initiated, high inhibitor concentrations were required to prevent continued pit growth. It was concluded that commercial corrosion inhibitors which are effective with respect to minimising generalised corrosion might not be as effective in minimising localised corrosion.

This paper focuses on evaluating the generalised and localised corrosion behaviour of carbon steel in the absence and presence of an imidazoline based corrosion inhibitor. In particular, we report on the influence of evaluating generalised and localised corrosion behaviour on the selection of the optimum dosage of the corrosion inhibitor at 80°C in a CO₂-saturated sodium chloride (NaCl) solution. We also report on the effects of short term pre-corrosion (prior to inhibitor addition). Although some published studies have considered both generalised and localised corrosion behaviour in evaluating inhibitor performance, such studies have mainly been performed using relatively low temperatures (25-55°C).

2.0 Experimental Procedure

2.1 Material preparation

X65 carbon steel coupons with diameters of 25 mm and thickness of 6 mm were machined from a stock bar. Insulated wires were soldered to the steel specimens prior to the steel being

embedded within a two component commercially available resin producing an exposed area of 4.9 cm² per test specimen The preparation process prior to each experiment consisted of wetgrinding each specimen successively with 120, 320 and 600 silicon carbide (SiC) grit paper. Specimens were then rinsed with acetone and distilled water before being dried using compressed air. For each experiment, two specimens were prepared 30 minutes prior to immersion into 1L of brine solution. The elemental composition of X65 carbon steel is provided in Table 1; the material has a ferritic-pearlitic microstructure.

С	Si	Mn	Р	S	Cr	Мо	Ni
0.12	0.18	1.27	0.008	0.002	0.11	0.17	0.07
Cu	Sn	A1	D	Nh	т:	X7	Ea
u	511	AI	D	ND	11	V	re

Table 1: Elemental composition of X65 steel (wt.%)

2.2 Solution preparation and chemical reagents

A 3 wt.% NaCl solution, prepared by dissolving analytical grade NaCl (Sigma-Aldrich) in deionised water, was used in all the experiments. The brine solution was prepared at least 24 h in advance of each experiment. CO₂ saturation was achieved by continuous CO₂ bubbling through the brine overnight as well as during each experiment. For experiments where the corrosion inhibitor was used, the inhibitor was injected using a calibrated micro-pipette. The inhibitor is an imidazoline synthesised by the reaction of tall oil fatty acid (TOFA) and diethylene triamine (DETA) (Figure 1).



Figure 1: The main components of imidazoline based DETA/TOFA corrosion inhibitor: imidazoline 1 and amide 2.

2.3 In situ electrochemical measurements

Linear polarisation resistance (LPR) measurements were acquired throughout the experiments to monitor the changes in general corrosion rate. The reciprocal of the polarisation resistance $(1/R_p)$ and the open circuit potential (OCP) were monitored with time. The three-electrode cell configuration consisted of the carbon steel specimen (working electrode) and a combined Ag/AgCl reference/platinum electrode which acted as the reference and counter electrode,

respectively. An ACM Gill 8 potentiostat was used to polarise the working electrode at ±15mV with respect to the OCP using a scan rate of 0.25 mV/s; we implemented a time interval of 15 minutes between successive LPR measurements.

2.4 In situ pH measurements

The bulk solution pH was measured at the start of each test, after 2 h of exposure (before inhibitor addition) and after 24h and 48h of exposure. The pH meter, a Thermo Scientific Orion Star A211 benchtop meter integrated with automatic temperature correction, was calibrated using pH 4, 7 and 10 buffer solutions prior to every experiment. For each pH measurement, the probe was immersed in the brine solution and equilibrated before recording the pH. pH measurements were taken for repeat experiments which enabled calculation of experimental error.

2.5 Experimental method

Two types of experiments were conducted to evaluate the performance of the imidazoline corrosion inhibitor, as shown in Figure 2. The first set of experiments was conducted to determine the optimum inhibitor dosage required to inhibit both generalised and localised corrosion. The second set of experiments was conducted to determine the effects of a short term pre-corrosion period at 80°C. A 2 h pre-corrosion period was chosen as it enabled the corrosion inhibitor to be tested on steel surfaces which had undergone corrosion however without any precipitation of crystalline corrosion products which could impact the optimum inhibitor dosage. Longer pre-corrosion periods can also have a major influence on the brine chemistry resulting in a high bulk pH which would not be representative of field conditions.



Figure 2: Outline of inhibitor performance experiments

2.5.1 Inhibitor performance

The inhibitor was tested at several different concentrations using the glass cell setup shown in Figure 3, both with and without a 2 h pre-corrosion period. For experiments with 2 h pre-corrosion, the pre-corrosion period began when the specimens were inserted into the test brine and the inhibitor was injected after 2 h. In the experiments without pre-corrosion, the specimens

were inserted into the test brine which already contained the inhibitor. The total testing time was 48 h for all the experiments. The brine solution was continuously bubbled with CO₂ throughout the experiments and a magnetic stirrer (100 rpm) was used to ensure continuous mixing of the solution.



Figure 3: Glass vessel set-up used for experiments

2.6 Surface analysis methods

2.6.1 Scanning electron microscopy (SEM)/energy dispersive X-ray spectroscopy (EDX)

A Carl Zeiss EVO MA15 VP SEM was used in conjunction with an Oxford instruments AZtecEnergy energy dispersive X-ray (EDX) system which provided both secondary and backscattered imaging while enabling elemental scans. The instrument was used to capture the top view images of the specimens. For top view imaging, the disc shaped specimens were coated with carbon along their circumference in order to minimise any surface charging effects. The system was set with a voltage range of 5-30 kV and a current range of 0.1-5 nA.

2.6.2 NP_{FLEX} 3D surface profilometry

NPFLEX 3D surface profilometry was used to determine the occurrence and extent of localised corrosion (pitting) on the carbon steel specimens. Before analysis, the post-test specimens were prepared by cleaning the surface using Clarke's solution in accordance with ASTM Standard G 1-03 [11] to ensure that no corrosion products were present. Two 3 x 3 mm² regions were scanned on two specimens for each experimental condition. The average of the 10 deepest pits present on four 3 x 3 mm² regions across two specimens was then used to determine the severity of the localised corrosion (maximum pit depth). Topographical analysis was achieved using a $2.5 \times$ objective with 3.5 mm working distance. The data was analysed using Vision64 software package.

3.0 Results

3.1 Performance of imidazoline corrosion inhibitor

The reciprocal of the polarisation resistance and the OCP measurements for experiments employing inhibitor concentrations in the range 0–30 ppm are shown in Figure 4; the data are plotted as a function of time. The reciprocal of R_p indicates that, in the absence of corrosion inhibitor, the corrosion rate is stable during the period 5–48 h. The corrosion inhibitor was injected after 2 h pre-corrosion. Even for the relatively low dosages of 10 and 20 ppm, there was a subsequent response in the form of a reduction in the corrosion rate, with $1/R_p$ decreasing from 0.017 and 0.015 ohm⁻¹cm⁻² to 0.007 and 0.004 ohm⁻¹cm⁻² (Figure 4 (a)) and OCP increasing from -690 mV to -650 and -625 mV (Figure 4 (b)), at 10 and 20 ppm respectively, after 48h. When the inhibitor concentration was 30 ppm, $1/R_p$ decreased more rapidly to a significantly lower value of 0.0003 ohm⁻¹cm⁻². In this case, we observe two 'waves' of OCP increase; firstly, the OCP increases to -635 mV and stabilises for 6 hours before increasing again, at a slower rate, to a maximum value of -600 mV. Such behaviour may indicate a two staged mechanism of adsorption/interaction of the inhibitor with the steel surface.

When the inhibitor concentration is \geq 40 ppm (Figure 5), $1/R_p$ decreases to values which are greater than those observed when the inhibitor concentration was 30 ppm, indicating a lower efficiency of inhibition of generalised corrosion (Figure 4 and 5 (a)). However, when using \geq 40 ppm inhibitor, the rate of reduction of $1/R_p$ was higher within the first 6 h, indicating a more rapid inhibitor film formation process.







Figure 4: (a) Reciprocal of polarisation resistance in ohm⁻¹cm⁻² and (b) open circuit potential in mV as a function of time determined for X65 carbon steel exposed to a CO₂saturated 3 wt.% NaCl solution at 80°C for a total experimental time of 48 h with imidazoline concentrations ranging from 0 to 30 ppm, added after 2 h of pre-corrosion





Figure 5: (a) Reciprocal of polarisation resistance in ohm⁻¹cm⁻² and (b) open circuit potential in mV as a function of time for X65 carbon steel exposed to a CO₂-saturated 3 wt.% NaCl solution at 80°C for a total experimental time of 48 h with imidazoline concentrations ranging from 40 to 100 ppm, added after 2 h of pre-corrosion

Figure 6(a) shows the pH of the bulk solution measured as a function of time in the same experiments. The initial pH was in the range 3.9–4.1 and, after pre-corrosion, the pH had risen to the range 4.3–4.5. After 48 h, the pH was in the range 4.8–5.5. The presence and concentration of the inhibitor determined the extent to which the pH of bulk solution increased from inhibitor addition to the end of the experiments. For example, 30 ppm inhibitor resulted in a final pH 4.8 compared to a final pH 5.5 for the uninhibited case. The final pH values are consistent in terms of the ranking of the corrosion rates based on electrochemical measurements (Figures 4 and 5).

Figure 6(b) shows inhibition efficiency as a function of inhibitor concentration. The end-point inhibitor efficiency was calculated from the polarisation resistance before the inhibitor was injected and the final polarisation resistance after 48 h of exposure (Equation 1). The integrated inhibitor efficiency was determined by integration of the polarisation resistance as a function of time to determine the overall efficiency of the corrosion inhibitor (Equation 2).

$$E_{Endpoint}(\%) = \left[1 - \left(\frac{R_{P \ Final}}{R_{P \ Initial}}\right)\right] \times 100 \qquad Eq. 1$$

$$E_{Integrated}(\%) = \left(1 - \left[\int_{2}^{48} \left(\frac{R_{P \ Final}}{R_{P \ Instantaneous}}\right)\right]\right) \times 100 \qquad Eq. 2$$

Where $R_{P \ Final}$ is the inhibited polarisation resistance at the end of the test (ohm.cm²) and $R_{P \ Initial}$ is the uninhibited polarisation resistance before inhibitor addition (ohm.cm²).

The end-point and integrated inhibitor efficiency data show similar trends with inhibitor concentration (Figure 6(b)). Again, we observe that the optimum inhibitor concentration (in terms of generalised corrosion) is 30 ppm. Inhibitor concentrations \geq 40 ppm result in a significant decrease in inhibitor efficiency relative to the optimum concentration.



Figure 6: (a) Bulk solution pH and (b) inhibitor efficiency for X65 carbon steel exposed to a CO₂-saturated 3 wt.% NaCl solution at 80°C for a total experimental time of 48 h with imidazoline concentrations ranging from 0 to 100 ppm, added after 2 h of pre-corrosion

Ex-situ scanning electron microscopy (SEM) analysis of the surfaces exposed to varying inhibitor concentrations (Figure 7) revealed that the X65 carbon steel had been substantially corroded in the absence of the corrosion inhibitor and no corrosion products appeared to have precipitated onto the steel surface after 48 h (Figure 7(a)). In the presence of 10 ppm inhibitor, two distinct areas were observed (Figure 7(b)). One of the areas appeared to be rougher and more severely corroded; this area was similar to the surfaces created without inhibitor. The surface created with 10 ppm inhibitor (Figure 7(b)) indicates heterogeneous corrosion due to incomplete coverage by the inhibitor film. With 30 ppm inhibitor and higher concentrations (Figure 7 (c and d)) the steel surface appeared smoother with very little corrosion, suggesting that at \geq 30 ppm the inhibitor provided improved protection due to near full coverage by the inhibitor film. These results are consistent with the data shown in Figure 6(b).



Figure 7: SEM images of X65 carbon steel exposed to a CO₂-saturated 3 wt.% NaCl solution at 80°C with imidazoline concentrations of (a) 0 ppm, (b) 10 ppm, (c) 30 ppm and (d) 50 ppm, added after 2 h of pre-corrosion for a total experimental time of 48 h

The exposed surfaces were also analysed using an NPflex surface profilometer to determine the extent of localised corrosion (Figure 8 (a)). After 48 h exposure, measured pit depths were in the range $23-26 \mu m$ when the inhibitor concentration was in the range 0-30 ppm. There was a sharp decrease in the maximum pit depth ($1.7 \mu m$) when the inhibitor concentration was increased to 40 ppm but higher concentrations of inhibitor (50-100 ppm) resulted in a slight increase in maximum pit depth. The reduction in maximum pit depth was used to calculate a localised inhibitor concentration in Figure 8 (b). This data shows that the optimum inhibitor concentration required to minimise localised corrosion is 40 ppm. These data underline the importance of evaluating both generalised and localised corrosion behaviour in selecting the optimum inhibitor concentration.



Figure 8: (a) Maximum pit depth and (b) Inhibitor efficiency comparison for X65 carbon steel exposed to a CO₂-saturated 3 wt.% NaCl solution at 80°C with imidazoline concentrations ranging from 0 to 100 ppm, added after 2 h of pre-corrosion for a total test duration of 48 h

The extent of localised corrosion in the presence of 30 or 40 ppm inhibitor is further compared in Figure 9. Localised corrosion is clearly seen in the presence of 30 ppm inhibitor whereas no pitting corrosion can be seen when the inhibitor concentration is increased to 40 ppm.



Figure 9: Surface profilometry images for X65 carbon steel exposed to a CO₂-saturated 3 wt.% NaCl solution at 80°C after 48 h in the presence of (a) 30 ppm and (b) 40 ppm imidazoline inhibitor which was added after 2 h of pre-corrosion

3.2 Effect of 2 h pre-corrosion on inhibitor performance

The effects of the 2 h pre-corrosion period on the efficiency of the inhibitor were evaluated (Figure 10). Changes in $1/R_p$ with time show that, without pre-corrosion, 30 ppm inhibitor forms a protective film more rapidly but the final film is less efficient than the film formed on the 2 h pre-corroded surface.

Without pre-corrosion, the OCP stabilised within the first 5 h of exposure, however with 2 h precorrosion a two-step increase in the OCP was observed (Figure 10 (b)) and the OCP took 25 h to stabilise. This indicates that the short term pre-corrosion period changes the inhibitor adsorption behaviour and, ultimately, this leads to a more efficient film and a lower $1/R_p$ is obtained.



Figure 10: (a) Reciprocal of polarisation resistance in ohm⁻¹cm⁻² and (b) open circuit potential in mV as a function of time for X65 carbon steel exposed to a CO₂-saturated 3 wt.% NaCl solution at 80°C for a total experimental time of 48 h with 30 ppm imidazoline with and without 2 h of pre-corrosion

The pH data for the 30 ppm inhibitor experiments, with and without a 2 h pre-corrosion period, are compared to pH data without inhibitor in figure 11. Without pre-corrosion, the more rapid film formation evidenced by monitoring $1/R_p$ with time results in a lesser change in the pH during the same period. There is a greater increase in pH within the first 2 h of exposure in the presence of a 2 h pre-corrosion period as the steel substrate is able to freely corrode. There is also a more significant increase in bulk pH after 2 h of exposure for the 2 h pre-corroded steel specimen which can be explained by the slower adsorption kinetics (Figure 10). However once the reciprocal of polarisation resistance stabilises for the 2 h pre-corroded specimen, it is lower than that observed in the absence of a pre-corrosion period (Figure 10) resulting in a greater increase in bulk pH between 24 to 48 h of exposure for specimens which have not undergone a pre-corrosion period (Figure 11).



Figure 11: Bulk solution pH for X65 carbon steel exposed to a CO₂-saturated 3 wt.% NaCl solution at 80°C for a total experimental time of 48 h with 30ppm imidazoline with and without 2 h of pre-corrosion

A top-view SEM comparison of the steel surfaces exposed to 30 ppm inhibitor in the absence/presence of the 2 h pre-corrosion period (Figure 12) showed that in the absence of precorrosion (Figure 12 (a)) there were two distinct areas on the steel surface. One area was more heavily corroded than the other suggesting that the inhibitor had adsorbed onto the steel faster on some areas compared to other. In contrast, the specimen that had undergone 2 h of precorrosion (Figure 12 (b)) appeared to be homogeneously protected by the inhibitor.



(a)

(b)

Figure 12: SEM images of X65 carbon steel exposed to a CO₂-saturated 3 wt.% NaCl solution at 80°C for a total experimental time of 48 h with imidazoline concentrations of 30 ppm (a) without pre-corrosion (b) with 2 h of pre-corrosion for a total experimental time of 48 h

As shown in Figure 13(a), generalised corrosion inhibitor efficiencies (end-point and integrated calculations) indicate that the 2 h pre-corrosion period resulted in an increase in inhibition efficiency from 86 and 93% to 98 and 97% for end-point and integrated efficiencies, respectively. These results are inconsistent with results gathered by other authors [6-8] however this can be attributed to the length of the pre-corrosion period which, in this case, was not long enough for corrosion products to precipitate. However, further analysis of the localised corrosion behaviour (Figure 13 (b)) revealed that although the 2 h pre-corrosion period had improved inhibition of generalised corrosion, localised corrosion had worsened which is consistent with literature [6].

As a result of the 2 h pre-corrosion period, the maximum pit depth had increased from 1.2 to 22.8 μ m after 48 h of exposure which meant that the inhibitor efficiency with respect to localised corrosion had dropped from 95.4% to 12.7%. The results indicate that short term pre-corrosion in the absence of corrosion products may result in improved efficiency of the corrosion inhibitor with respect to the uniform corrosion loss, however it could also lead to a diminution in protection afforded against localised corrosion. Surface profilometry images of the pits (Figure 14(a)) confirmed that there was no sign of localised attack on the steel surface in the absence of any pre-corrosion. However, on the steel surface exposed to 30 ppm of the corrosion inhibitor after a 2 h pre-corrosion period there was severe localised corrosion attack (Figure 14(b)).



Figure 13: (a) Uniform corrosion inhibitor efficiency and (b) maximum pit depth for X65 carbon steel exposed to a CO₂-saturated 3 wt.% NaCl solution at 80°C with 30 ppm imidazoline with and without 2 h of pre-corrosion for a total experimental time of 48 h



Figure 14: Surface profilometry 2D images for X65 carbon steel exposed to a CO₂saturated 3 wt.% NaCl solution at 80°C with 30 ppm imidazoline for a total experimental time of 48 h (a) without pre-corrosion and (b) with 2 h of pre-corrosion

4.0 Conclusions

Experiments were performed to quantify the performance of an imidazoline inhibitor in terms of its ability to minimise generalised and localised corrosion of X65 carbon steel immersed in CO_2 -saturated sodium chloride brine for 48 hours at 80°C.

Our data show that due consideration of both generalised and localised corrosion behaviour is necessary in order to select the optimum inhibitor dosage. A short term (2 hour) pre-corrosion period was found to improve the efficiency of the corrosion inhibitor with respect to the uniform corrosion behaviour however at the cost of promoting localised corrosion attack.

5.0 Data availability

The raw/processed data required to reproduce these findings cannot be shared at this time as the data also forms part of an ongoing study.

6.0 References

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