

# An Electrochemical and Raman Investigation of Guanine as an Environmentally Friendly Corrosion Inhibitor for API 5L X65 Steel in HCl Solutions

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Guanine was investigated as a potential green corrosion inhibitor to API 5L X65 carbon steel in HCl 0.1 mol.dm<sup>-3</sup> solution, at pH = 2.0, under hydrodynamic conditions using electrochemical, weight loss and Raman spectroscopy experiments. Cyclic voltammetry and potentiodynamic polarization curves suggested that Guanine does not change the metal/solution interface appreciably and weight loss tests confirmed the low inhibition efficiency (IE) of Guanine in protecting the X65 steel in this corrosive medium. An IE = 22% was determined after 48 h of exposure, a value lower than that reported in the literature on the mild steel/Guanine/HCl system. Raman spectroscopy was employed to gain insight on the nature of the steel/Guanine interaction and the results pointed out to a weak physisorption of the molecule, thus explaining the very low IE values obtained.

**Keywords:** Guanine, API 5L X65 steel, HCl solution, green corrosion inhibitors.

## 1. Introduction

Corrosion of metallic materials is a ubiquitous problem in many industries and its efficient control is required to ensure the safety of the operations, prevent downtime, human life losses and environmental hazards<sup>1-3</sup>.

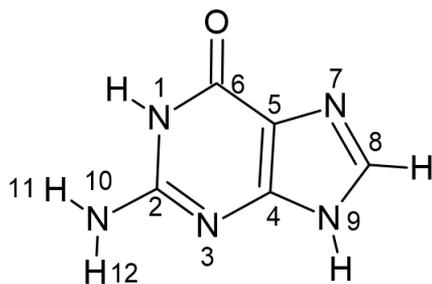
The API 5L X65 steel is a high strength low alloy carbon steel widely employed in the manufacture of pipelines for the oil and gas industry due to its excellent mechanical properties and low cost<sup>4-8</sup>. However, during service, this material is exposed to highly aggressive environments that may contain acid gases, such as carbon dioxide and hydrogen sulfide, elevated chloride concentrations and low pH combined with high temperature and pressure conditions. An example of such harsh conditions is the operation of acidification of wells aimed at improving the oil production rates, where the steel is exposed to high concentrations of hydrochloric acid (HCl)<sup>4-8</sup>. As the X65 steel is prone to corrode in such media, corrosion inhibitors are usually added to avoid metal dissolution. The inhibitors most widely used in the oil and gas industry are amines, aldehydes, mercaptans, nitrogen heterocycles and other compounds containing sulfur<sup>9-11</sup>. Due to the growing ecological awareness and the adoption of stricter environmental regulations, greener alternatives to inhibitor formulations containing toxic compounds, such as chromate, mercaptans or benzotriazoles, are required<sup>11</sup>.

In this context, imidazole and its derivatives are often tested as low toxicity inhibitors for carbon steel and other metallic materials<sup>12-24</sup>. Our research group has been part of this

ongoing effort, using a methodology that unites electrochemical and gravimetric tests with Raman spectroscopy to achieve a better understanding about the metal/inhibitor interaction both from a macro and microscopic viewpoint<sup>22-24</sup>.

Among the derivatives tested, Guanine (Figure 1) is one of the most interesting, since it is a purine nitrogen base that composes DNA and RNA nucleic acids, and therefore is omnipresent in living organisms and the environment<sup>24</sup>. This molecule is also encountered in cosmetics and food products<sup>25</sup>. Its nontoxicity combined with multiple adsorption sites and low cost make it a suitable candidate to a green corrosion inhibitor. Despite these characteristics, there are very few reports on the use of Guanine as corrosion inhibitor, with the most recent contribution being made by our group<sup>24</sup>. Guanine was tested on copper in 0.1 mol.dm<sup>-3</sup> HCl solutions, pH = 2.0, where electrochemical and weight loss experiments revealed that it has an inhibition efficiency (IE) of 87% at its highest concentration (0.001 mol.dm<sup>-3</sup> or 150 ppm). Surface Enhanced Raman Spectroscopy (SERS) showed that the protonated Guanine (GuanineH<sup>+</sup>), which was the main species at pH = 2.0, deprotonates upon adsorption, leaving the neutral molecule chemisorbed on the copper surface via Cu-N<sub>3</sub> coordination. Eventually, the copper electrode becomes covered with a thick and adherent passivating layer of Cu(Guanine)Cl compound, that protects the surface both at room and moderate temperatures (T = 60 °C)<sup>24</sup>. Tao et al.<sup>21</sup> found similar results concerning Guanine inhibition of copper in H<sub>2</sub>SO<sub>4</sub> solutions via electrochemical and weight loss experiments, where an IE = 92% was determined.

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**Figure 1.** The Guanine molecule structure and its atom numbering scheme.

After a thorough literature review only two papers addressing Guanine as corrosion inhibitor for carbon steel were found. Yan and coworkers<sup>19</sup> studied Guanine as inhibitor for mild steel in 1.0 mol.dm<sup>-3</sup> HCl solutions. Electrochemical and weight loss experiments led to an IE = 68% when 0.001 mol.dm<sup>-3</sup> Guanine was added to the solution and quantum chemical calculation results carried out on the isolated molecule suggested a physisorption through the  $\pi$ -electron cloud, but no spectroscopic technique was employed to probe the surface-inhibitor interaction. Chahul et al.<sup>20</sup> tested Guanine as inhibitor corrosion for mild steel in H<sub>3</sub>PO<sub>4</sub> 0.1 mol.dm<sup>-3</sup> solutions using weight loss and potentiodynamic polarization measurements. Their results pointed out that Guanine acts as a mixed type inhibitor with an IE = 69%, in close agreement with Yan et al.<sup>19</sup>. Fitting of the data to a Langmuir adsorption isotherm revealed that the molecule physisorbs on the metal surface, a result that was further corroborated by the reduced IE at higher temperatures (T = 333 K).

It is worth mentioning that neither of the investigations aforementioned employed spectroscopic techniques to get insight on the nature of the steel/inhibitor interaction. Our publications show how relevant is the use of spectroscopic information to better interpret the IE results<sup>22-24</sup>. Besides that, the tests were all carried out in stagnant conditions where the relevance of the mass transport of a heavily solvated molecule, such as Guanine, to the metal surface cannot be properly assessed. Furthermore, to the best of our knowledge, Guanine was not investigated as a possible corrosion inhibitor for X65 steel. Due to the relevance of this material to the oil and gas industry and to the growing necessity of green inhibitors, we believe that an investigation of the Guanine/X65 steel/HCl using a methodology that combines electrochemical and spectroscopic test carried out under hydrodynamic conditions is justified.

## 2. Experimental Methodology

### 2.1. Solution preparation

Polycrystalline powdered Guanine (98% purity) and concentrated HCl (37% m/v) were purchased from Sigma-Aldrich and Merck, respectively. These reagents were used without further purification, as they were already reagent grade.

The test solutions were HCl 0.1 mol.dm<sup>-3</sup>, at pH = 2.0, containing or not 0.001 mol.dm<sup>-3</sup> Guanine, other concentrations of the inhibitor were not tested because the literature agrees

well that this concentration provides the maximum IE<sup>18-21,24</sup> and it is also the solubility limit of Guanine in the HCl solution at the test pH. For the Guanine solutions, the needed amount of the substrate for a solution of  $c = 0.001$  mol.dm<sup>-3</sup> was weighted and completely dissolved in the required volume of concentrated HCl. Deionized water was then added to complete 1 dm<sup>3</sup> under vigorous stirring. Then the pH was adjusted to 2.0 using concentrated HCl or NaOH as needed. The solution containing both HCl and Guanine will be henceforth only referred to as the Guanine solutions.

### 2.2. Electrochemical experiments

The working electrode was made of a X65 rod machined to match a rotating disc electrode (RDE) apparatus and embedded in epoxy resin, leaving a circular area of 0.160 cm<sup>2</sup> exposed to the solution. All electrochemical tests were carried out in a standard three electrode cell, using a saturated calomel electrode (SCE) and a platinum grid as reference and counter electrodes, respectively.

Before immersion, the working electrode was mechanically cleaned using a polisher with emery papers of different mesh up to 600 mesh. They were then washed with deionized water, sonicated with acetone for degreasing and dried in air flow.

Cyclic voltammetry and potentiodynamic polarization experiments with both solutions started after stabilization of the open circuit potential (OCP). The cyclic voltammetry had its cycles beginning at  $E_1 = -0.6$  V to avoid evolution of hydrogen gas with a sweep rate of 0.01 V/s, from  $E_2 = +0.3$  V to  $E_3 = -1.0$  V. For the polarization curve experiments, the sweep rate was of 0.000125 V/s, starting from  $-0.3$  V and ending on 0.5 V vs OCP. All experiments were made in triplicate, at room temperature. All the potentials are quoted to the SCE scale and a rotation speed of 1000 rpm was employed in the electrochemical tests. An IVIUM CompactStat potentiostat was used in the tests to acquire the electrochemical data. Test results were processed using the IVIUM and Origin Pro 8.5 softwares.

### 2.3. Weight loss experiments

Weight loss experiments were performed by immersion of test coupons in either HCl 0.1 mol.dm<sup>-3</sup> or Guanine 0.001 mol.dm<sup>-3</sup> solution, pH = 2.0, at room temperature (298 ± 2 K) for 48 h. The solutions were stirred at 1000 rpm, using stirring plates and a magnetic bar. Prior to immersion, rectangular samples of API 5L X65 steel of 12 – 14 cm<sup>2</sup> of area were prepared in the same way as the RDE for the electrochemical tests. The ratio of solution volume to specimen area was kept at 30 – 40 mL.cm<sup>-2</sup> to minimize pH changes during immersion time and the tests were stopped as soon as any pH changes were detected. After immersion, the samples were cleaned in the appropriate solution, dried and weighed. The cleaning procedure was repeated until constant weight and all measurements were made in triplicate, according to ASTM G1<sup>26</sup>.

### 2.4. Raman and infrared measurements

A Bruker SENTERRA confocal Raman microscope was used to measure the Raman spectra of both solid guanine and the X65 steel samples after immersion for the weight loss experiments. The 532 nm excitation line was used with

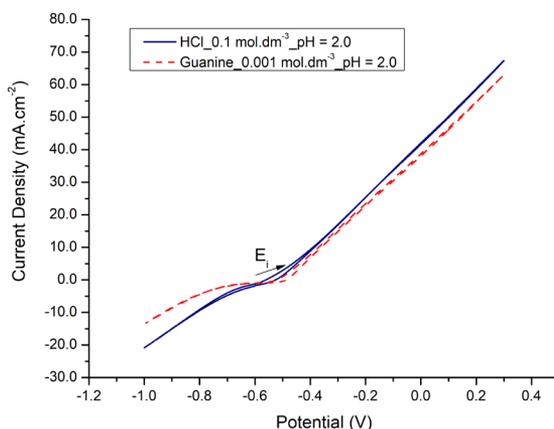
10 mW of power focalized on the sample by a 20x long working distance objective (Olympus). The samples were accommodated in a microscope slide and three coadditions of 30 s were collected with 3 - 5  $\text{cm}^{-1}$  resolution.

Infrared spectra were obtained on a VERTEX 70 model infrared spectrometer (Bruker) in the attenuated total reflection (ATR) mode, using a diamond crystal. Each spectrum was acquired with 4  $\text{cm}^{-1}$  resolution and 64 scans were considered enough to achieve a good signal to noise ratio.

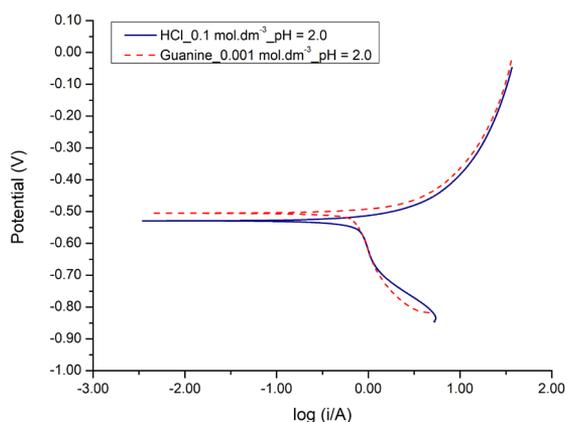
### 3. Results and Discussion

Cyclic voltammograms for the HCl 0.1  $\text{mol}\cdot\text{dm}^{-3}$  and Guanine 0.001  $\text{mol}\cdot\text{dm}^{-3}$  solutions, at pH = 2.0, are shown in Figure 2. It is possible to observe that the cycles obtained for both solutions are quite similar, showing that this molecule is not able to modify the steel/solution interface in a meaningful way. Indeed, both curves show a continuous increase in current density as the applied potential becomes more positive, showing that the iron is continually being dissolved as  $\text{Fe}^{+2}$ , which is the corrosion product expected for tested conditions, according to the Pourbaix diagram of the Fe/ $\text{H}_2\text{O}$  system<sup>27</sup>. The current density decreases gradually after the inversion potential ( $E = +0.3$  V) until the OCP (-0.54 V) is reached. After that, the current density decreases again due to reduction of the  $\text{H}^+$  ions until the end of the experiment, at  $E = -1.0$  V. Addition of Guanine causes only a minor reduction of the maximum anodic current density, that drops from 67  $\text{mA}\cdot\text{cm}^{-2}$  in the voltammogram of the blank solution to 62  $\text{mA}\cdot\text{cm}^{-2}$  in the presence of this molecule. Besides that, one can see a small current plateau in the region between -0.5 and 0.7 V and a decrease in the current density observed at  $E = -1.0$  V in the cathodic sweep of the cycle obtained for the Guanine solution, indicating that the purine makes the hydrogen evolution reaction a little more difficult to take place. The lack of new features in the voltammogram of the inhibited solution suggest that formation of a new Guanine-iron compound did not happen during the experiment, perhaps because of its small-time scale.

Potentiodynamic polarization was also carried out for the X65 steel RDE immersed in either HCl 0.1  $\text{mol}\cdot\text{dm}^{-3}$  or Guanine 0.001  $\text{mol}\cdot\text{dm}^{-3}$ , at pH = 2.0 and 1000 rpm. The curves obtained for both solutions can be seen in Figure 3. Inspection of the curves reported in Figure 3 show that the electrochemical behavior of the steel electrode is very similar in both solutions, in strict agreement with cyclic voltammetry results. The slope of the curves also showed no significant difference, meaning that a protective layer was not formed on the X65 steel electrode. In previous experiments carried out using a copper electrode, a peak at  $E = 0.0$  V was clearly seen, followed by a current plateau, a behavior that is remarkably different from the steel electrode. Besides that, at the end of the experiment, a passive layer could be clearly seen at the copper electrode surface. The absence of significant changes in the polarization curves of the X65 steel electrode in both solutions and the very similar aspect of generalized corrosion of the RDE surface after the test in both media make it reasonable to anticipate that Guanine is not an efficient inhibitor to X65 steel in HCl solutions under hydrodynamic conditions.



**Figure 2.** Voltammograms of a X65 steel RDE immersed in a HCl 0.1  $\text{mol}\cdot\text{dm}^{-3}$  solution (solid blue line) and a Guanine 0.001  $\text{mol}\cdot\text{dm}^{-3}$  solution (dashed red line), pH = 2.0, at 1000 rpm.



**Figure 3.** Potentiodynamic polarization curves for the neat HCl 0.1  $\text{mol}\cdot\text{dm}^{-3}$  solution (solid blue curve) and Guanine 0.001  $\text{mol}\cdot\text{dm}^{-3}$  solution (dashed red curve), pH = 2.0, at 1000 rpm.

The electrochemical results point to a very limited corrosion inhibition activity from Guanine bound to the time frame of those experiments. Therefore, the weight loss experiments were conducted to investigate whether the inhibition efficiency of Guanine could improve in greater immersion times. As a matter of fact, its IE increased with exposure time in the experiments with the copper RDE<sup>24</sup>.

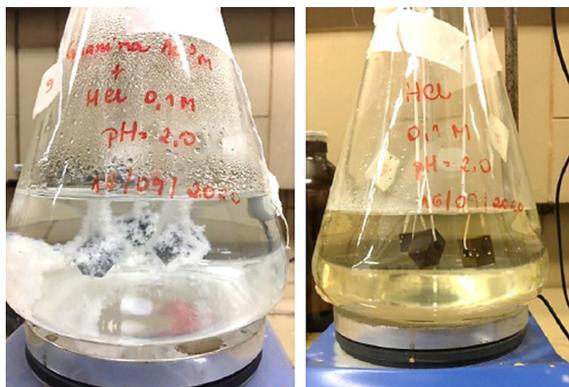
The influence of the immersion time on the IE of Guanine toward X65 steel was probed through the gravimetric tests. Visual inspection of the solutions and coupons during the test showed that after 24 h of immersion, the solutions were nearly the same as shortly after the immersion and the coupons were nearly equally attacked, while the pH of both solutions was stable, but after 48 h of immersion, significant changes could be observed. The coupons immersed in the HCl solution was covered with a dark brown layer, characteristic of oxidation, and the ones exposed to the Guanine solution were less darkened than the ones immersed in the blank solution and small white flakes loosely adhered to the metal could be seen together with a greater amount of the same flakes at bottom of the flask. The test was immediately stopped as

the pH of the blank solution increased from 2.3 to 5.9 and that of the Guanine solution changed from 2.1 to 3.3. The aspect of the solutions and the coupons after the test can be seen in Figures 4 and 5, respectively.

The aspect of the coupons after the immersion test as well as the final pH of the solutions suggested that corrosion occurred in a smaller extent in the test carried out in the presence of Guanine. Corrosion rates and weight loss values were calculated to verify this possibility and the results obtained are shown in Table 1.

**Table 1.** Average weight loss per area, corrosion rates and inhibition efficiency (IE) values calculated after 48 h of immersion of X65 steel coupons in solutions of either HCl 0.1 mol.dm<sup>-3</sup> or Guanine 0.001 mol.dm<sup>-3</sup> (initial pH = 2.0).

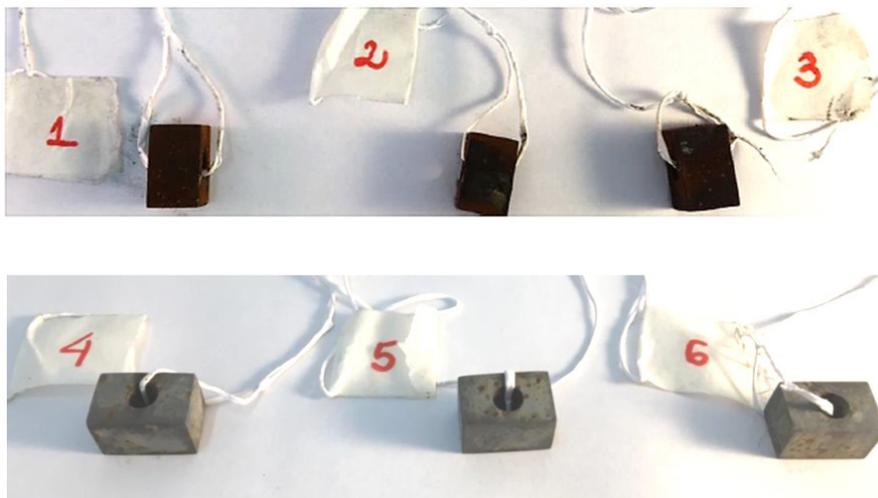
Solution	Average Weight Loss/Area (g.cm <sup>-2</sup> )	Average Corrosion Rate (mm.year <sup>-1</sup> )	Inhibition Efficiency (%)
HCl 0.1 mol.dm <sup>-3</sup>	8.5795	2.0074	-
Guanine 0.1 mol.dm <sup>-3</sup>	6.6540	1.5569	22.44



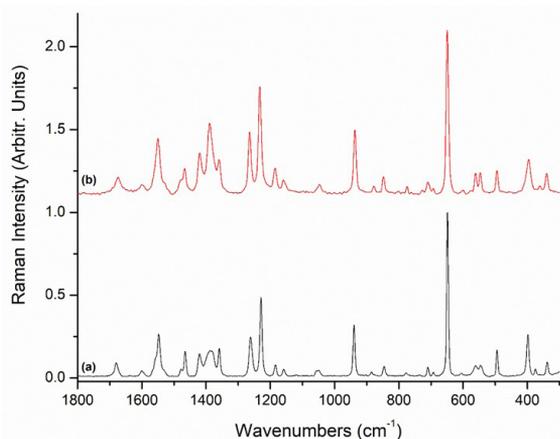
**Figure 4.** Weight loss test after 48 h of immersion, before withdrawal of the X65 steel coupons. Guanine solution test – left panel. HCl solution test – right panel.

The very high values of mass loss per area and corrosion rates obtained for the Guanine solution resulted in an IE of 22%, thus revealing that this molecule is a really poor inhibitor for X65 steel in the tested media, corroborating the results anticipated by the electrochemical experiments. As the final pH of the Guanine solution was still quite acidic, formation of soluble Fe<sup>2+</sup> ions took place almost freely since formation of oxide layers that could partially hinder the migration of the aggressive ions from the solution does not take place at this pH. Besides that, the flaky white compound did not adhere to the surface, so it did not have a passivating effect. As a matter of fact, if Guanine is able to interact with the steel surface, such interaction must be very weak. Finally, it is worth mentioning that the IE of Guanine calculated in this work is even smaller than the ones found by Yan et al.<sup>19</sup> for mild steel in spite of the lower HCl concentration employed in this work. The lower IE found here may be justified by the use of a relatively high rotation speed while the other authors used stagnant conditions. Additionally, the much smaller IE of Guanine in the protection of X65 than that obtained for copper in the same experimental conditions can be explained by the absence of an adherent and insoluble protective film of an Fe(II)-Guanine complex, as was observed for copper (Cu(I)GuanineCl complex)<sup>24</sup>.

Raman spectroscopy was employed to characterize the steel/inhibitor interaction. The spectrum was acquired on a steel coupon immersed for 48 h in the 0.001 mol.dm<sup>-3</sup> Guanine solution and can be seen in Figure 6, where the spectrum of a solid Guanine sample was added for the sake of comparison. To the best of our knowledge, this is the first report on a surface spectrum acquired on steel exposed to Guanine solutions. The reason why only normal Raman spectra were acquired on steel rather than the much more sensitive Surface Enhanced Raman Spectroscopy (SERS) is that iron is not a good substrate to SERS, since it is easily oxidized and cannot sustain the surface plasmon resonance, which is the main signal enhancement mechanism, in the conditions used in this work<sup>28</sup>. However, analysis of the spectra in Figure 6 provides useful insight on the nature of the steel/Guanine interaction.



**Figure 5.** Photographs of the coupons after 48 h of the gravimetric test. Coupons 1 to 3 were immersed in the HCl 0.1 mol.dm<sup>-3</sup> solution while coupons 4 to 6 were exposed to the 0.001 mol.dm<sup>-3</sup> Guanine solution.



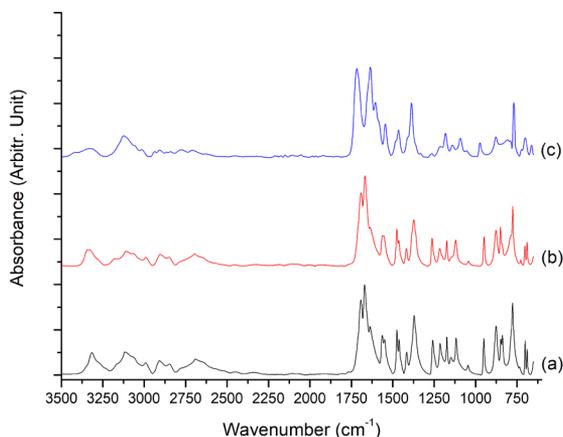
**Figure 6.** Raman spectra of (a) solid guanine and (b) X65 coupon that was immersed in Guanine solution for 48 h.

It is easily observed that the spectrum acquired on the metal surface is very similar to that of the solid Guanine, used in the preparation of the solution. When a molecule interacts strongly with a metal surface, its Raman spectrum differs from that of the isolated species due to perturbation of the local molecular environment, changes in geometry and coordination, which reflects on the bonds force constants. The stronger the interaction the greater are the observed changes. Since the spectra of the metal surface exposed to Guanine and that of the neat compound are nearly the same, it can be assumed that the molecule interacts very weakly with the steel, most likely through a very weak physisorption. The very low IE derived from the gravimetric tests agrees with this result. Such a behavior is fundamentally different from that observed with copper, where Guanine chemisorbs on the metal surface as the first step to form an adherent polymeric Cu(I)GuanineCl complex that blocks the surface efficiently.

Finally, characterization of the white flaky product formed during the weight loss experiment with X65 steel was carried out using infrared spectroscopy. Figure 7 shows the acquired spectrum, together with spectrum of neat, powdered Guanine and the Cu(I)Guanine complex, whose characterization is still underway. A close look to Figure 7a and 7b reveals that the spectra of the white product and that of Guanine have the same spectral pattern and the band positions are very similar, allowing to conclude that the white product formed on X65 steel is actually Guanine that precipitated from the solution due to the increase in the pH, which resulted in lower solubility. Comparison with the spectrum of the Cu(I)Guanine complex makes it clear that an Fe-Guanine was not formed and once again corroborates the low IE of Guanine toward X65 steel protection in HCl media.

#### 4. Conclusions

Electrochemical tests carried out for the X65 steel immersed in  $0.1 \text{ mol}\cdot\text{dm}^{-3}$  HCl solutions containing or not  $0.001 \text{ mol}\cdot\text{dm}^{-3}$  Guanine under hydrodynamic conditions revealed that this molecule is not able to modify the metal surface in a meaningful way, a fact that was latter confirmed by weight loss experiments where an inhibition efficiency of 22% was derived. Further corroboration of the very weak



**Figure 7.** Infrared spectra of (a) Guanine, (b) the white flaky product formed during weight loss experiments in the X65 steel/Guanine/HCl system and (c) Cu(I)Guanine complex.

steel/Guanine interaction was given by the Raman spectra acquired directly on the metal surface after immersion in the solution containing Guanine. Infrared spectroscopy revealed that the flaky product formed on the X65 steel coupons during the weight loss experiments were Guanine that precipitated from the solution due to pH induced solubility changes and not an insoluble non-adherent Fe-Guanine complex.

A weak physisorption mechanism is proposed based on that, in agreement with previous studies performed with mild steel. The very low IE of Guanine precludes any practical applications as corrosion inhibitor for the studied system, despite its nontoxicity. The very weak interaction of Guanine with steel is the main reason for its much lower IE when compared with copper under the same experimental conditions.

#### 5. Acknowledgments

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