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# Effects of 4-bromoaniline and pentylamine compounds on the stress corrosion cracking of mild steel and brass in dilute electrolyte solutions

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**Abstract.** The inhibition effect of 4-bromoaniline (4BA), pentylamine (PTL) and their combined admixture on the stress corrosion behaviour of mild steel and brass in 0.5 M and 0.3 M H<sub>2</sub>SO<sub>4</sub> and NH<sub>4</sub>OH solution was studied through open circuit potential measurement and visual observation. The experiment was performed at 90% stress level for optimal results. Results obtained showed that the inhibitor compounds generally influenced the thermodynamic tendency of the alloys to corrode, delayed the onset of crack formation and specimen failure in the electrolytes. The combined admixture of 4BA and PTL performed most effectively in the both acid solutions, increasing the corrosion potential to -0.391 V and -0.281 V compared to the corresponding values obtained in the control acid solutions at -0.446 V and -0.451 V. 4BA performed most effectively in NH<sub>4</sub>OH solutions with corrosion potential values of -0.743 at 504 h. Visual observation of the metallic alloys shows crack initiated on mild steel in both acids without inhibitor addition at 72 h of exposure and specimen failure occurred at 216 h. The corresponding observation for brass in 0.5 M NH<sub>4</sub>OH solution showed crack initiated at 144 h before failure at 360 h. In 0.3M NH<sub>4</sub>OH, crack initiation and failure occurred at 216 h and 504 h respectively. The admixture of 4BA and PTL delayed crack initiation on mild steel to 216 h and no specimen failure was observed at 504 h. 4BA delayed the onset of crack formation on brass in 0.5 M and 0.3 M NH<sub>4</sub>OH solution to 216 h and 288h. However, failure occurred at 432 h in 0.5 M NH<sub>4</sub>OH compared to no failure at 504 h of exposure in 0.3 M NH<sub>4</sub>OH solution.

**Keywords:** stress corrosion cracking; inhibitor; 4-bromoaniline; pentylamine

## 1. Introduction

Corrosion of metallic alloys is a naturally occurring phenomenon wherewith they degrade progressively to the lowest possible energy states through chemical and or electrochemical interaction with other compounds [1, 2]. Most structural alloys corrode gradually due to atmospheric exposure and presence of moisture in air [3, 4]. Other chemical degrade the alloys relatively rapidly due to the presence of highly reactive corrosive anions. Being a reaction controlled mechanism, corrosion initiates and propagates on material surfaces [5-7]. Techniques to hinder the electrochemical mechanisms responsible for corrosion on metallic surfaces enhances their corrosion resistance. There are corrosion reaction processes that are insidious and nearly impossible to detect making it unpredictable. The probability of metals to undergo electrochemical reaction with O<sub>2</sub>, H<sub>2</sub>O, chlorides, sulphates etc. in aqueous environment is one of the major factors responsible for corrosion [8, 9]. Destruction by corrosion strongly depends on (i) surface properties of the metal or alloy, (ii) presence of flaws, impurities and materials within the microstructure of the alloys (iii) composition of the corrosive electrolyte (iv), oxide scales (which may be continuous or broken) etc. Corrosion is the most common cause of structural failure to equipment, parts and structures in industry. Corrosion is costly to maintain coupled with the burdensome economic, societal, design, health, safety and technological repercussions. In the United States, the effect and consequence of corrosion domestic appliances, house maintenance and public infrastructure is approximately \$22 billion which by extension impacts the financial sector of the economy [10]. In 2002, the US Federal Agency carried out a



study which concludes that the direct costs of metallic corrosion on U.S. industries totals \$276 billion [10]. Mild steel is the most economical of all steels in terms of cost and the most commonly applied in industry for a significant number of industrial machineries and devices. It is weld-able, very durable, recyclable and readily available. Most tools, equipment, components and machineries encountered today is produced from mild steel such as vehicle chassis, radiators, automobile joints and linkages etc. The weak corrosion-resistance of mild steel necessitates the use of paint and other coatings to prevent rust formation. In most cases coating from oil or grease significantly hinders rust formation. Brass is an alloy of copper and zinc with high degree of recyclability [11]. It is extensive domestic application due to its aesthetic value. Brass is also employed in applications where friction must be minimized such as in locks, gears, bearings, doorknobs, ammunition casings and valves. The acoustic properties of brass enable its application in the production of musical instruments. [12]. Chemical compounds that alter the mechanism of electrochemical reactions on steel surfaces thus hindering corrosion have been proven to be effective. These compounds generally known as corrosion inhibitors can be organic or inorganic [13-18]. In contribution to the research on possible corrosion inhibitors, this article focusses on the study of 4-bromoaniline and pentylamine on the corrosion inhibition of mild steel and brass in dilute  $H_2SO_4$  and  $NH_4OH$  solution.

## 2 Experimental methods

Mild steel (MS) and brass (BRS) specimens were purchased from the open market in Lagos in the form of cylindrical pipes with in standard size with NPS designator number 1.5. Spectroscopic analysis of the alloys is shown in Table 1 below;

Table 1: Spectroscopic analysis of BRS and MS

<b>BRS</b>											
<b>Element</b>	Cu	Zn	Fe	Pb	Others						
<b>% Wt.</b>											
<b>Content</b>	61	35.5	0.35	2.5	0.63						
<b>MS</b>											
<b>Element</b>	Fe	C	Si	P	S	Cr	Ni	Mn	Cu	Zn	Ca
<b>% Wt.</b>											
<b>Content</b>	99.4	0.13	0.028	0.007	0.018	0.008	0.025	0.307	0.035	0.022	0.017

The outer diameter of the brass and mild steel being 50.1mm (1.9724 in.) and 50.9mm (2.0039 in.) with thickness of 5mm (0.19724 in.) and 0.405mm (0.15945 in.) respectively. The alloy specimens were machined with lathe machine to the designed dimensions. The metals were metallographically prepared with respect to the ASTM G38 before stressing of the specimens. The C-ring is a specimen type specifically prepared to determining experimentally the susceptibility of a material to stress corrosion cracking (SCC). Three different stress levels were experimented on in the control acid and base solution to identify the most appropriate stress level to use. The equation below was employed to determine the stress applied to a C-ring specimen:

$$OD_f = OD - \Delta, \Delta = \frac{f\pi D^2}{4Etz}$$

where OD represents the outside diameter of C-ring before stressing, in. (or mm),  $OD_f$  represents the outside diameter of stressed C-ring in. (or mm),  $f$  indicates the desired stress in MPa (or psi) (within the proportional limit),  $\Delta$  represents the change of OD giving desired stress, mm (or in.),  $D$  represents the mean diameter ( $OD - t$ ), mm (or in.),  $t$  is the wall thickness, mm (or in.),  $E$

indicates the modulus of elasticity, MPa (or psi) and  $z$  is the correction factor for curved beams [11].

Determination of  $OD_f$  for (a) Ms and (b) BRS is shown below;

**(a)**

$$E = 14.5 \times 10^6$$

$$f = 61200 \text{ MPa (90\% of 6000 psi)}$$

$$OD = 1.9724 \text{ in., } t = 0.19685 \text{ in.}$$

$$D = 1.77555 \text{ in., } z = 0.92$$

$$\Delta = \frac{61200 \times \pi \times 1.77555^2}{4 \times 14.5 \times 10^6 \times 0.15945 \times 0.92}$$

$$OD_f = 1.9724 - 0.05771$$

$$OD_f = 1.91469 \text{ in. (48.6mm)}$$

**(b)**

$$E = 30 \times 10^6$$

$$f = 54000 \text{ MPa (90\% of 6000 psi)}$$

$$OD = 2.0039 \text{ in., } t = 0.15945 \text{ in.}$$

$$D = 1.84445 \text{ in., } z = 0.94$$

$$\Delta = \frac{54000 \times \pi \times 1.84445^2}{4 \times 30 \times 10^6 \times 0.15945 \times 0.94}$$

$$OD_f = 2.0039 - 0.03209$$

$$OD_f = 1.97181 \text{ in. (50.08mm)}$$

Analar grade  $H_2SO_4$  and  $NH_4OH$  solutions were each prepared in molar concentration of 0.5 M and 0.3 M by dilution in predetermined quantity of deionized  $H_2O$ . 4-bromoaniline (4BA) and pentylamine (PTL) were both obtained from Sigma-Aldrich, USA. 4BA was grounded to powdery form and added to the prepared dilute acid and base solutions after heating the solution to about  $70^\circ C$  to obtain a perfect solution as the 4BA compound is insoluble in cold water. Volumetric concentration of 18.75% of 4BA was added into both acid and base concentrations. PTL crystals were grounded, measured (using Oarhus digital measuring scale) and added directly into the dilute acid and base solutions at volumetric concentration of 18.75%. A third set of acid and base inhibitor solutions were prepared with the combined admixture of 4BA and PTL at 18.75% concentration in 0.5 M and 0.3 M  $H_2SO_4$  and  $NH_4OH$  solutions. MS specimens were immersed in both the 0.5M and 0.3M  $H_2SO_4$  solution with and without the inhibitor compounds while BRS specimens were immersed in both the 0.5M and 0.3M  $NH_4OH$  solution with and without the inhibitor compounds. The Alloys were immersed in the electrolyte for 21 days and were checked every 24 h determine SCC initiation, crack growth and failure in the acid inhibitor solution. The half-cell potential was carried out using a multi-meter. Set at 20mA, probes are attached to the meter ports (neutral and working to the metal and the screw respectively). Copper/Copper-sulphate was used as the half-cell electrode. Readings were taken on a daily basis and recorded. Graphs were plotted of potential measurement taken in millivolts against time.

### 3 Results and discussion

#### 3.1 Stress analysis experiments

Generally, the result of the half-cell potential measurement carried out revealed the different events that compose the SCC life cycle which occurs in three stages namely initiation, growth and failure. The graphs show the values obtained from comparison of the different stress levels to determine the correct stress to apply. Results from the graphs show that 90% stress value was the best stress value to use in the experiments and as such the calculations used were based on such values.

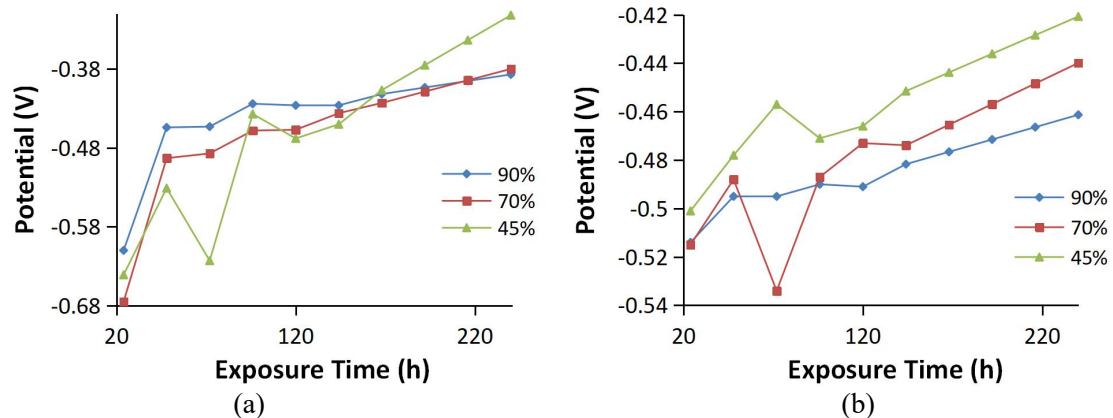


Figure 1: Stress analysis of MS in (a) 0.5 M  $\text{H}_2\text{SO}_4$  solution and (b) 0.3 M  $\text{H}_2\text{SO}_4$  solution

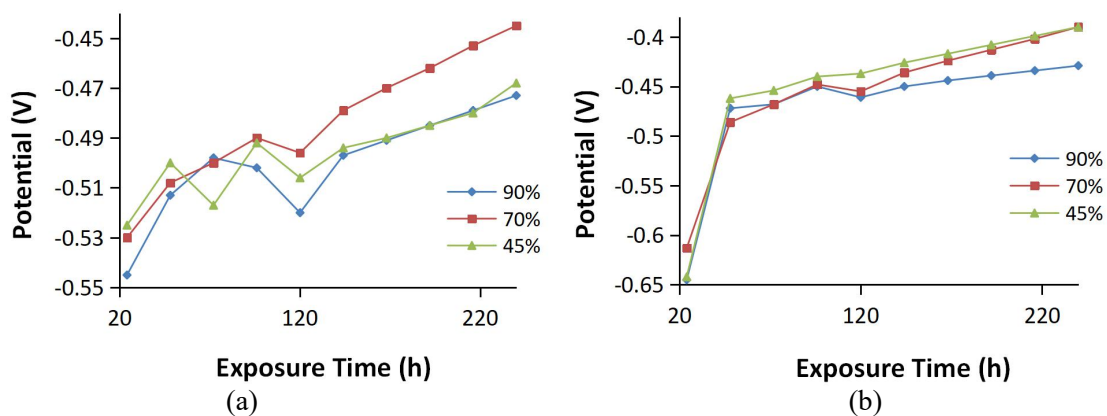


Figure 2: Stress analysis of BRS in (a) 0.5 M  $\text{NH}_4\text{OH}$  solution and (b) 0.3 M  $\text{NH}_4\text{OH}$  solution

### 3.2 Open circuit potential (OCP) measurements

The OCP plots of corrosion potential versus exposure time for MS in 0.5 M and 0.3 M  $\text{H}_2\text{SO}_4$  solution at specific concentrations of 4BA, PTL and admixture of 4BA and PTL compounds, and BRS in 0.5 M and 0.3 M  $\text{NH}_4\text{OH}$  solution at specific concentrations of 4BA, PTL and admixture of 4BA and PTL compounds are shown from Fig. 3(a) to Fig. 4(b). The plots show the relationship between corrosion potential resulting from electrochemical action occurring on the alloys surfaces subjected to 90% stress level. Observation of Fig. 3(a) and (b) shows the OCP plots at various inhibitor concentrations in the acid solution generally shifted in the positive direction with respect to exposure time till 504 h. However, there were portions where the plots alternated between positive and negative potentials due to alteration of the electrochemical processes where the corrosion rate of MS fluctuates. The plots at 0% inhibitor concentration was the most electronegative due to the absence of inhibitor molecules to counteract the aggressive action of corrosion species on the steel. Fig. 3(a) shows the plots from the combined admixture of 4BA and PTL was the most electropositive followed by the plot from 4BA. In Fig. 3(b), the plot from the combined admixture of 4BA and PTL was also the most electropositive by a wide margin followed by the plot from PTL. These observations show acid concentration influences the performance of inhibitor compounds. The plots in Fig. 4(a) and (b) significantly contrast the observations in Fig. 3(a) and (b). The plot configurations generally shifted to the negative direction signifying increase corrosion of BRS due to electrochemical action of the corrosive species in the base solution. While the transition to lower corrosion potentials was more visible and significant in Fig. 4(b) after 24 h of exposure, brief active-passive transition occurred on Fig.

4(a) till 192 h of exposure before progressing negatively till 504 h. In 0.5 M and 0.3 M  $\text{NH}_4\text{OH}$  solution [Fig. 4(a) and (b)], 4BA compound proves to be the most effective inhibitor compound being the most electropositive at 504 h followed by the combined admixture of 4BA and PTL in Fig. 4(a) and PTL in Fig. 4(b).

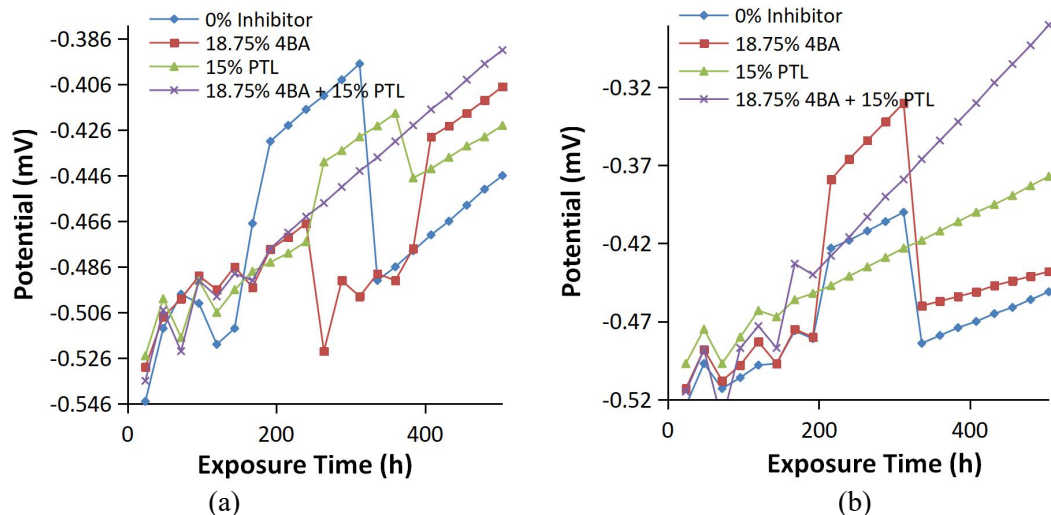


Figure 3: OCP plots of MS in (a) 0.5 M  $\text{H}_2\text{SO}_4$  solution and (b) 0.3 M  $\text{H}_2\text{SO}_4$  at specific concentrations of 4BA, PTL and combined admixture of 4BA and PTL

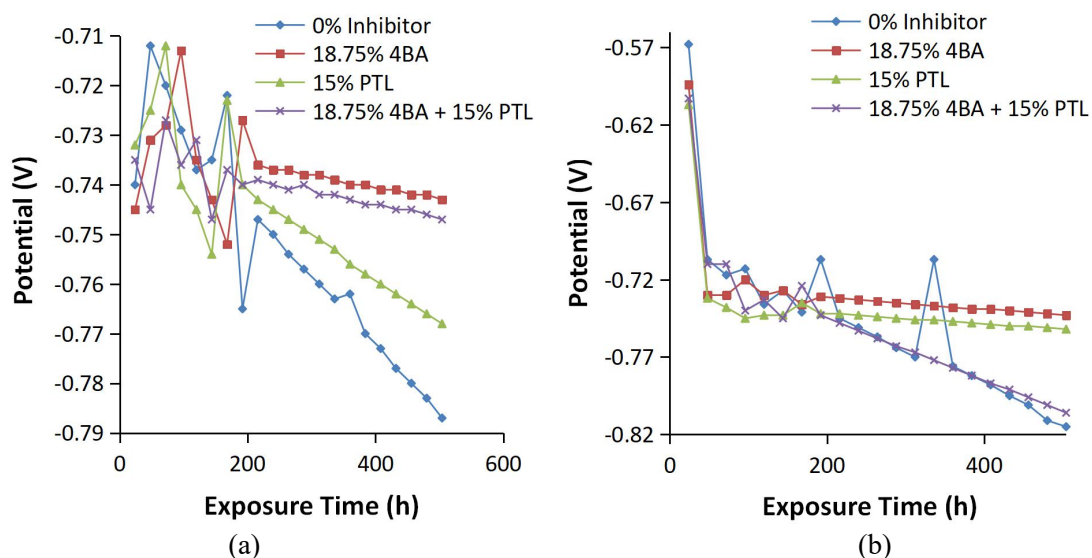


Figure 4: OCP plots of MS in (a) 0.5 M  $\text{NH}_4\text{OH}$  solution and (b) 0.3 M  $\text{NH}_4\text{OH}$  at specific concentrations of 4BA, PTL and combined admixture of 4BA and PTL

### 3.3 SCC Observations of control MS and BRS specimens

Observation of SCC phenomena in 0.5M  $\text{H}_2\text{SO}_4$  solution without any inhibitor addition showed the presence of crack initiation before 72 h of exposure which grew progressively until failure occurred at 216 h of exposure. Before the onset of crack formation, aggressive corrosion reaction mechanisms occurred due to the oxidation of the steel a result of the electrochemical action of  $\text{SO}_4^{2-}$  ions in the acid solution. General corrosion occurred over the exposed steel until 72 h. In 0.3 M  $\text{H}_2\text{SO}_4$  solution SCC cracks appeared at 72 h of exposure. Between 144-216 h of exposure,

hairline crack growth propagated along the bent radius of the steel. Beyond 216 h of exposure, SCC failure of the steel occurred. SCC observation of BRS in 0.5 M  $\text{NH}_4\text{Cl}$  solution contrasts the earlier observation of MS in its control acid media. Cracks initiated at 144 h after undergoing severe redox electrochemical processes on its surface. Growth of the crack was observed at 216 h until several other cracks branched out in different directions from the initial cracks at 288 h. At 360 h of exposure BRS failed. In 0.3 M  $\text{NH}_4\text{Cl}$  solution SCC cracks also occurred on BRS. However, there was no visible corrosion reaction observed until 216 h where brown coloration of the alloy was prevalent and SCC crack visible. Crack growth did not occur until 288 h with few corrosion pits. Branched cracks occurred at 360 h before failure at 504 h of exposure.

### 3.4 Corrosion of MS in the acid/inhibitor solution

Contrary to SCC crack initiation which occurred on control MS in 0.5 M  $\text{H}_2\text{SO}_4$  solution at 72 h, SCC crack initiated at 144 h in the presence of 4BA at 18.75% concentration due to the inhibiting action of the inhibitor. Crack growth occurred at 216 h and multiple cracks occurred at 288 h leading to failure at 504 h. In control 0.5 M  $\text{H}_2\text{SO}_4$  solution failure occurred at 216 h signifying improved operational lifespan of the steel. In 0.5 M  $\text{H}_2\text{SO}_4$ /PTL solution at 15% PTL concentration, SCC failure was delayed to 216 h with multiple corrosion pits visible on the stressed region of the steel. Hairline cracks propagated and branched out in different directions. However, there was no failure of the steel specimen after 504 h of exposure. Similar observation occurred in 0.5 M  $\text{H}_2\text{SO}_4$  solution in the presence of combined admixture of 4BA and PTL inhibitor compounds (18.75% 4BA and 15% PTL concentration). Comparing the observations to the performance of MS in 0.3 M  $\text{H}_2\text{SO}_4$  solution, crack initiated early at 72 h despite the presence of 4BA while growth progressed at 144 h till 288 h. Beyond 288 h branched cracks became visible until failure occurred at 360 h. Contrary to the poor performance of 4BA on MS in 0.3 M  $\text{H}_2\text{SO}_4$  solution, PTL delayed crack formation till 288 h while crack growth occurred at 360 h. Branched cracks became visible at 432 h but there was no failure signifying better performance compared to performance of 4BA. The combined admixture of 4BA and PTL compound performed similarly to the performance of PTL on MS in 0.3 M  $\text{H}_2\text{SO}_4$  solution.

### 3.5 Corrosion of BRS in the base/inhibitor solution

Observation of SCC of BRS in 0.5 M  $\text{NH}_4\text{OH}$  at 18.75% 4BA concentration shows 4BA delays the onset of crack initiation to 216 h compared to 144 h in 0.5 M  $\text{NH}_4\text{OH}$  without 4BA. Crack growth occurred at 288 h. Corrosion pits was also observed which were sustained till 432 h were failure of BRS occurred. In the presence of 4BA in the base solution BRS failed at a later hour compared to its performance without 4BA. However, PTL performed poorly in delaying the onset of SCC on BRS compared to 4BA. While crack initiated at 216 h in the presence of 4BA, crack initiated at 144 h in the presence of PTL and failure occurred at 360 h compared to 432 h with 4BA. 4BA delayed the onset of SCC cracks on BRS in 0.3 M  $\text{NH}_4\text{OH}$  to 288 h compared to 216 h in 0.5 M  $\text{NH}_4\text{OH}$ . Crack growth occurred but there was no failure observed after 504 h of exposure. Crack signs were also visible on BRS in the presence of PTL inhibitor at 216 h of exposure. PTL also hindered specimen failure of BRS at 504 h of exposure. The synergistic effect of 4BA and PTL in 0.5 M and 0.3 M  $\text{NH}_4\text{OH}$  showed that the combined admixture of both compounds had little influence on the SCC resistance of BRS in 0.5 M  $\text{NH}_4\text{OH}$  solution. However, in 0.5 M  $\text{NH}_4\text{OH}$ , crack initiation was delayed to 360 h and as at 504 h of exposure no failure was observed.

## Conclusion

Stress corrosion crack growth of mild steel and brass, and its inhibition with 4-bromoaniline, pentylamine and their admixture was studied by open circuit potential measurement and visual detailed surface inspection in dilute  $\text{H}_2\text{SO}_4$  and  $\text{NH}_4\text{OH}$  environments. 4-bromoaniline was most

effective on brass in  $\text{NH}_4\text{OH}$  environment delayed crack initiation and hindering specimen failure compared to control brass specimen without inhibitors which crack very early during exposure and eventually failed under 90% stress limit. Corrosion potential of the inhibited brass significantly contrast the potential obtained for control brass due to alteration of the electrochemical mechanisms on brass by the inhibitor molecules. Admixture of 4-bromoaniline and pentylamine compounds performed most effectively on mild steel in  $\text{H}_2\text{SO}_4$  solution with generally similar results obtained for brass.

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