

# PLANT EXTRACTS AS CORROSION INHIBITORS FOR ALUMINIUM PIGMENTS IN ALKALINE MEDIUM

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## Abstract

The inhibition effects of some plant extracts on the corrosion of aluminium pigments in alkaline medium was investigated using a gas volumetric technique at pH 8.5 and 10.5 and temperatures 30 and 40°C. The studied plant materials include *Dacryodes edulis* (DE), *Mucuna pruriene* (MP), *Pentacletra macrophylla* (PM), *Caladium bicolor* (CB), *Newbouldier laevis* (NBL). The gas volumetric method enabled determination of the latency period of the aluminium pigment which represents the time required to initiate the corrosion reaction in the medium. Inhibition efficiency was determined by comparing the latency periods in absence and in presence of the plant extracts. The results obtained indicate that all the extracts inhibited the corrosion reaction in the reaction medium with inhibition efficiency (%) of 95.4, 96.6, 98.0, 98.5, 99.0 for NBL, CB, MP, PM and DE respectively at 5.0 %(v/v) inhibitor concentration.

**Keywords:** Aluminium pigments, plant extracts, corrosion inhibition, alkaline medium.

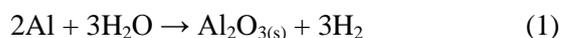
## 1.0 Introduction

Aluminium pigments are used in printing and silver inks, as decorative topcoats for metallic structures (frames, gates, doors, railings, masts, posts and other constructions or machine parts) and as a means for protecting the metallic surfaces against corrosion (Kiehl and Greiwe 1999; Karlson *et al*, 2006; Lui *et al*, 2007). The protective action of Al coatings is based on the fact that, all aluminium surfaces form a dense, almost impermeable oxide layer on the surface upon exposure to air (Wheeler, 1999).

Aluminium pigments are usually manufactured from aluminium granules by a ball milling process in the presence of mineral spirit and fatty acid (Karlson, 2006). During the process of forming, the surface areas of the aluminium particles are drastically increased, resulting in high surface reactivity by water (Karlson, 2006; Kiehl and Greiwe 1999). Due to the high reactivity of Al pigment particles with water, aluminium coatings have traditionally been solvent borne (Muller *et al*, 2001a, 2001b; Karlson *et al*, 2006; Chattopadhyay, 2001). However, due to increasing awareness about the environment and ensuing strict environmental regulations governing emission of organic solvents, solvent

borne aluminium coatings are giving way to water borne coatings

It has been observed that, under the slightly alkaline conditions in which most water-borne paints exist, aluminium reacts with water to form aluminium oxide and aluminium hydroxide according to the scheme (Karlson, 2006; Lui *et al*, 2007; Kiehl and Greiwe, 1999; Reisser *et al*, 1995):



These reactions lead to loss of the shiny aluminium appearance of the pigment particles and more importantly the evolved hydrogen gas may lead to unacceptable pressure build up in containers and constitute explosion hazards (Lui *et al*, 2007; Muller, 2002, 2004; Besold *et al*, 1991). Therefore, there is need to protect the surface of pigment particles using suitable corrosion inhibitors which show sufficient affinity for the pigment surface. Again, owing to increasing ecological awareness and strict environmental regulations as well as the inevitable drive towards sustainable and environmentally friendly processes, attention is now focused on the



development of substitute nontoxic alternatives to inorganic and organic inhibitors applied earlier. Consequently the current focus in corrosion inhibitor research is to identify and develop new classes of non toxic, benign, inexpensive and efficient alternatives. In this regard, there has been increasing interest in investigating natural products of plant origin for corrosion inhibiting efficacy.

Abdel-Gaber *et al* (2008) investigated the inhibition of aluminium corrosion in alkaline solutions using *Ambrosia maritime L* (damsissa) extract employing different chemical and electrochemical techniques. Priya *et al.* (2005) studied the corrosion behaviour of aluminium in rain water containing garlic extract. The inhibitive action of the acid extracts of seeds, leaves and barks from the *Ficus virens* plants towards hydrochloric and sulphuric acid corrosion of aluminium has been tested using weight loss and thermometric techniques (Jain *et al.*, 2006). Kliskic *et al* (2000) analyzed aqueous extract of *Rosmarinus officiantus* as corrosion inhibitor for aluminium alloy corrosion in chloride solution. EL-Hosary *et al* (1972) studied the corrosion inhibition of aluminium and zinc in HCl using *Hisbiscus subdariffa* extract. Avwiri and coworkers (2003) studied the inhibitive action of *Vernonia amygdalina* on the corrosion of aluminium alloy in HCl and H<sub>2</sub>SO<sub>4</sub>. Several studies from our laboratory investigated the corrosion inhibition efficiency of some plant extracts including *Sansevieria trifasciata*, *Ocimum basilicum*, *Telfaria occidentalis* extracts on aluminium and mild steel corrosion in different environments using the gas volumetric technique (Oguzie *et al.*, 2005; Oguzie, 2007 and 2008).

Unlike aluminium metal, corrosion and corrosion inhibition of aluminium pigments has not received as much attention. Nevertheless, the few studies in the area have demonstrated that the same theories and mechanisms apply and some organic compounds that inhibit aluminium metal corrosion also function well on aluminium pigments. The organic inhibiting agents applied so far include phenols and aromatic acids, alkyl phosphates and other anionic amphiphiles (Karlsson *et al.*, 2006; Das *et al.*, 2004; Kummert *et al.*, 1980; Muller *et al.*, 2001a, Muller 2002). A common feature for all of them is of that they contain functional groups that interact specifically

with aluminium at the surface by adsorption and plant extracts are also rich in such species.

The present report assesses the suitability of plant extracts as corrosion inhibitors for aluminium pigments and investigates the inhibiting effect of leaf extracts of *Newbouldia laevis* (NBL), *Caladium bicolor* (CB), *Pentaclethra macrophylla* (PM), *Mucuna pruriene* (MP) and *Dacryodes edulis* (DE) on the corrosion of aluminium pigments in aqueous alkaline environments using the gas volumetric technique. These plants have been chosen from literature survey based on their known medicinal and antimicrobial activities [Ekanem *et al.*, 2013; Mishra and Wagner, 2006, Ugbogu and Akukwe, 2009; Usman and Osuji, 2007]. However, they have not been previously utilized for the purpose of inhibiting aluminium pigment corrosion.

## 2.0 Materials and method

### 2.1 Materials

100% unstabilized nonleafing aluminum pigment powder (average particle diameter 16  $\mu\text{m}$  and specific surface about of 5m<sup>2</sup>/g: BET method) was used. The reagents include, butyl glycol, a wetting agent (adduct of 10 moles of ethylene oxide to nonylphenol), triaminomethylphenol (AMP) for pH adjustment. All reagents are of analytical grade and used as supplied by Benda Lutz of Austria.

### 2.2 The plant extract

Fresh green leaves from the plants of NBL, CB, PM, PM and DE used as inhibitors for this study was obtained from Ihiaqwa in Owerri West Local Government Area of Imo State, Nigeria. The leaves were sun dried to constant weight and latter ground into powder using an electric blender. Stock solutions of the extracts were prepared by boiling 100g of the ground leaves in 500mL of 0.5M NaOH for 3 hours (under reflux). The resulting solutions were allowed to cool to room temperature and then filtered to remove the plant residues. The amount of plant material extracted into solution was quantified by comparing the weight of the dried residue with the initial weight of the powdered plant material before extraction. The concentration of the stock solution was calculated in terms of v/v%.

### 2.3 The Gasometric Assembly

This is essentially an apparatus that measures the volume of gas evolved from a reaction system and consist of a 250mL Erlenmeyer flask connected via a delivery tube to a burette which in turn is connected to a reservoir of paraffin oil. This set-up provides a rapid and sensitive means for gauging any perturbation by an inhibitor with regard to gas evolution within the system. With regard to practical applications of aluminium pigments in waterborne paints the hydrogen volume evolved after a certain period of time is the most important measured quantity.

### 2.4 Experimental procedure

The corrosion medium was a mixture of 100ml of water and butyl glycol in the ratio of 9:1. To improve the wetting of the hydrophobic aluminium powder, 2.0 wt% of a wetting agent was added. The pH of the solution was raised to 8.5 and 10.5 with AMP. Then, 1.0g of the aluminium pigment powder was dispersed in the medium with a magnetic stirrer for about 5 min. The settled aluminium pigment of uninhibited dispersion showed initially a metallic sparkle which reacted by rapid effervescence resulting from hydrogen gas evolution to form aluminium hydroxide [Muller, 1999]. The progress of the corrosion reaction was monitored at 30 and 40°C respectively and the time required for the evolution of 100ml of H<sub>2</sub> gas. The volume of hydrogen gas evolved by the corrosion reaction was estimated by the volume change in the level

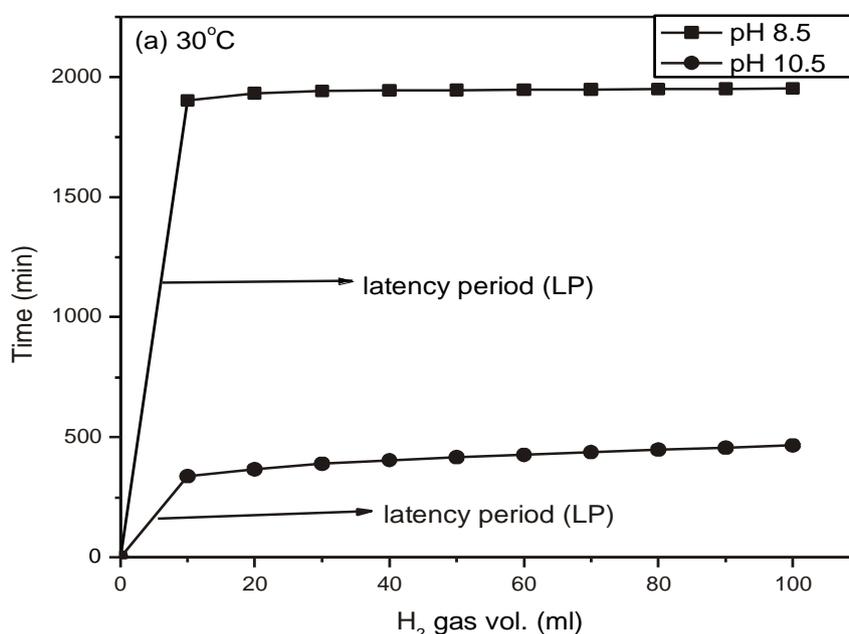
of the paraffin oil in the burette. The examined plant extracts were dissolved in concentration of 5.0%(v/v) in 100mL of the corrosion medium before the dispersion of aluminium pigment powder and the pH adjusted again as necessary. The progress of the corrosion reaction was monitored as before to obtain the time required for the evolution of 100ml of H<sub>2</sub> gas for each plant was determined.

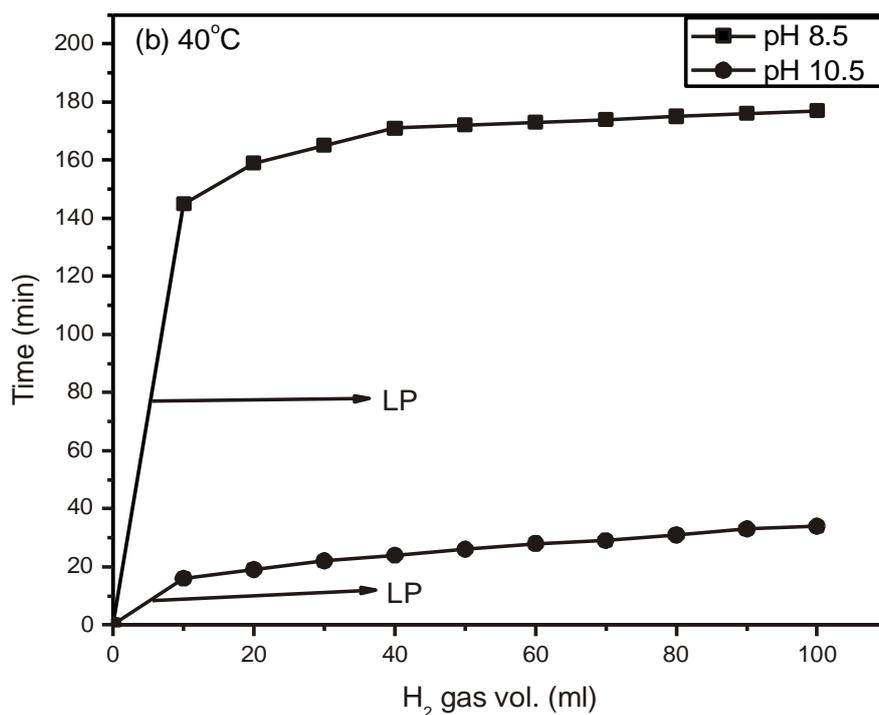
## 3.0 Results and discussion

### 3.1 Corrosion of aluminium pigments

The progress of the corrosion reaction of the standard aluminium pigments in inhibited and uninhibited systems was assessed by measuring the volume of hydrogen gas evolved with time and determining the time interval required for generation of 100 ml of hydrogen gas [Muller *et al.* 2001a]. The relative rapidity and effectiveness of the gas-volumetric technique as well as its suitability for monitoring in situ, any perturbation by an inhibitor with respect to gas evolution in metal/corrosion systems have been well established [Onuchukwu, 1988; Oguzie *et al.*, 2006; Oguzie, 2007]

Figures 1a and 1b present the gas-volumetric plots for the aluminium pigment in the uninhibited test solution at pH 8.5 and 10.5 and temperatures 30 and 4°C respectively. The plots depict the time interval required for the different systems to evolve fixed volumes of hydrogen gas (up to 100mL).



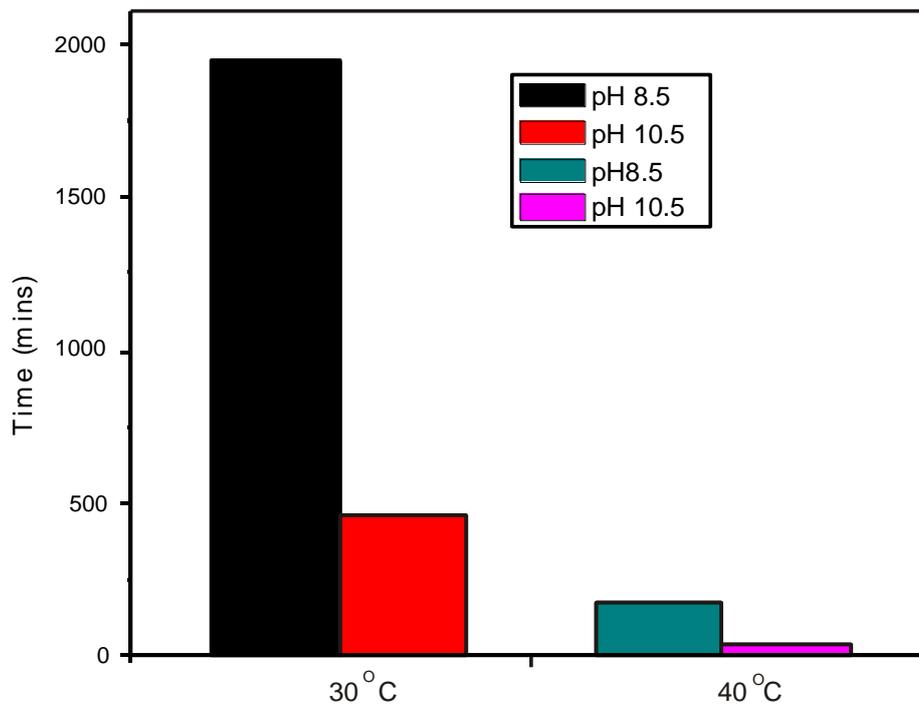


**Figure1.** Plots of the hydrogen gas evolution during the corrosion of standard aluminium pigment in alkaline medium at (a) 30°C and (b) 40°C.

Inspection of the plot reveals not only the presence of a period in which there were no gas evolutions but also a difference in the values obtained at pH 8.5 and 10.5. This period is referred to as the induction or incubation period (Oguzie, 2008) or latency period (Muller, 1999) and represents the time required by the solution to neutralize the pre-immersion oxide coating on the aluminium pigment surface. Similar pH-dependencies of the hydrogen evolution have been observed previously by other authors (Muller *et al*, 1994; Muller, 2004) and were attributed to the influence of the isoelectric point (IEP) of aluminium oxide (pH 9.0) (Karlson, 2007; Shriver *et al* 1994). At pH 8.0 (below the IEP of aluminium oxide) the aluminium oxide surface is positively charged, hence more of the negatively charged extract anions are adsorbed whereas above the IEP (at pH 10.0) aluminium oxide

surface is negatively charged considering the fact that this solution contains an abundance of negatively charged ions which can lead to electrostatic repulsion. This pH-dependency can also be explained by considering the stability of the aluminium surface as a function of pH derived from the Pourbaix diagram (Harman, 1994). At low pH Al is not stable in aqueous solutions and the formation of Al-ions will take place and at high pH aluminate anions are formed. Between pH 4.0 and 8.5 the oxide layer is stable which explains the longer latency periods at pH 8.5

Figure 2 compares the latency period obtained at pH 8.5 and 10.5 at 30°C with the latency period obtained at pH 8.5 and 10.5 at 40°C respectively. The lower latency periods at pH 10.5 at the higher temperature is indicative of the higher corrosion susceptibility of aluminium pigment at higher temperature [Muller *et al*, 2001b].



**Figure 2: Variation of latency periods with temperature during the corrosion of aluminium pigment at pH 8.5 and 10.5**

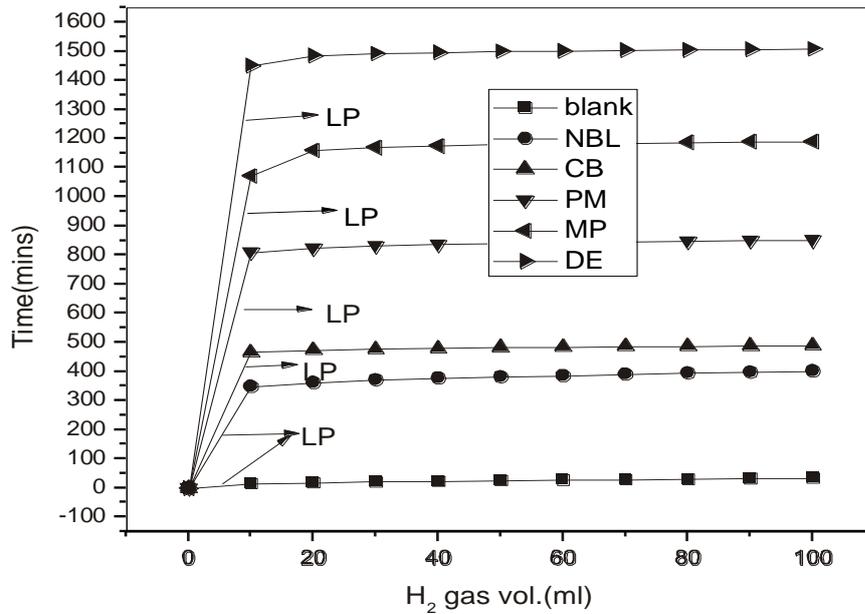
### 3.2 Addition of the plant extracts

All gas-volumetric results of the corrosion of the aluminium pigments at 40°C and pH 10.5 without and with addition of 5.0% (v/v) of NBL, CB, PM, MP and DE plant extracts. Figure 3 represents the gas evolution obtained for the different plants. The results at 30°C and pH 10.5 are provided in the section on ninety days experiment. Higher temperature and pH accelerates the corrosion reaction and is often better suited for a fast differentiation of corrosion inhibiting effects [Muller *et al.*, 2001a]. Longer latency periods were observed in the presence of the plant extracts which represents not only the time to break down the pre-immersion oxide but also the time to cause the desorption of the inhibitor molecules from the aluminium surface. After the latency period, the corrosion reaction

progressed with a high rate. The rate of hydrogen evolution could be approximated using the following equation:

$$V = kt \tag{3}$$

where V is the volume of the evolved H<sub>2</sub> gas at time t and k is the specific rate constant. This rate is similar beyond the latency period for each extract because once the inhibitor film is removed the underlying substrate is the same and should have similar properties. The values of the specific rate constant for each plant are given in Table 1 which shows the all plant extracts inhibited the corrosion rate of the aluminium pigment powder with DE as the most effective.

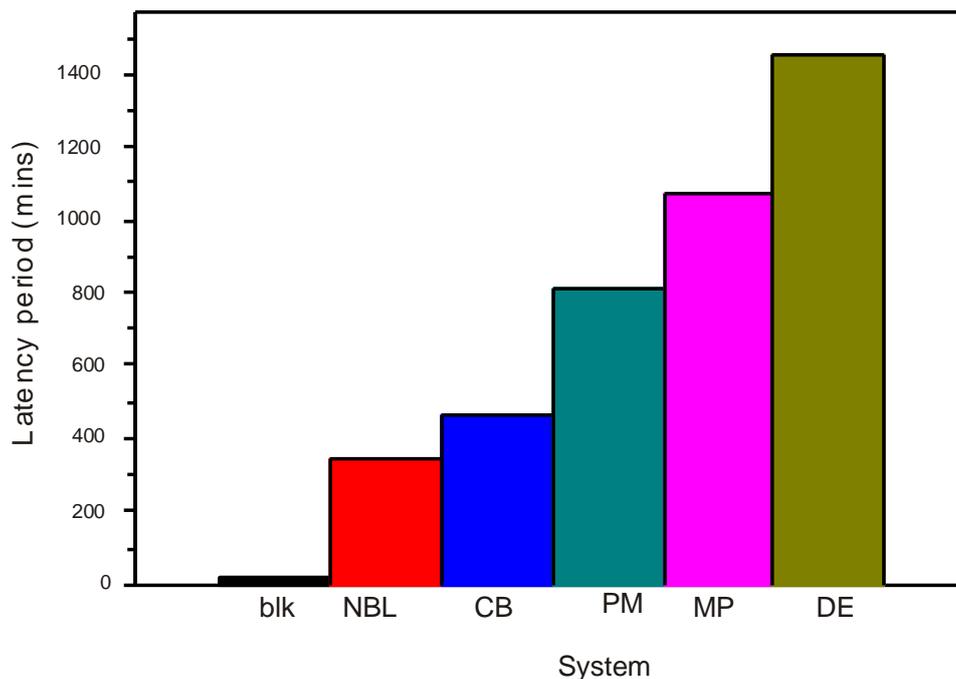


**Figure 3: Variation of hydrogen evolution with time during corrosion of aluminium pigments in alkaline medium at 40°C and pH 10.5 without and with the addition of (5.0g/L) plant extracts**

**Table 1: Values of the specific rate constant for the different plants**

Plant	blank	NBL	CB	PM	MP	DE
K (ml/min)	2.91	0.287	0.214	0.124	0.09	0.07

Figure 4 compares the latency periods of the inhibited solutions with the standard. The longer latency periods in the inhibited experiments are clear indications that the plant extracts inhibited the corrosion of the aluminium pigment in the corrosion medium.



**Figure 4: Comparison of the latency periods from the dispersions of the aluminum pigment at 40°C and pH 10.5 without and with addition of 5.0 g/L of NBL, CB, PM, MP and DE extracts**

### 3.3 Inhibition efficiency

The protective action of an organic compound during corrosion of a material is based on the adsorption ability of their molecules where the resulting adsorption film isolates the material from the corrosion medium [Oguzie, 2007]. Consequently in inhibited solutions, the corrosion rate is indicative of the number of free sites remaining after some sites have been effectively blocked by the adsorbed inhibitor [Oguzie, 2007]. If it is assumed that corrosion occurs only at the free sites while the covered sites have negligible corrosion rates the degree of surface coverage ( $\theta$ ) and hence the inhibition efficiency (IE%) can be calculated as follows [Muller, 1999]:

$$\theta = 1 - \frac{L_{free}}{L_{inh}} \quad (4)$$

$$IE\% = 1 - \left( \frac{L_{free}}{L_{inh}} \right) \times 100 \quad (5)$$

where  $L_{free}$  and  $L_{inh}$  correspond to the latency periods in the absence and in the presence of inhibitor respectively. The inhibition efficiencies values obtained using equation 5 is presented in Table 2.

**Table 2: Latency periods and inhibition efficiency (EI%) data for NBL, CB, PM, MP and DE**

System	Latency periods (mins)	IE%
blank	16	0
NBL	349	95.4
CB	467	96.6
PM	810	98.0
MP	1072	98.5
DE	1451	99.0

These results show that the longer the latency period the higher the inhibition efficiency. It is obvious that the additives displayed remarkable protective ability in the reaction medium which can be attributed to chemical composition of the plant extracts which includes tannins, saponins, carbohydrates, proteins etc. [Ekanem *et al*, 2013; Mishra and Wagner, 2006; Usman and Osuji, 2007] some of which have been reported to retard metal dissolution [Oguzie, 2008].

Attempt was also made to establish the trend of the inhibition efficiencies of each extract on the pigment surface as a function of the volume of hydrogen evolved within a given time interval. The result obtained is presented in Table 3 and shows all five plants extracts as excellent corrosion inhibitors for aluminium corrosion in alkaline medium. The slight variations are the result of slight variations in chemical composition of the different plants. The trend of the data suggests that the efficiency of the extracts gradually diminished as more hydrogen gas is evolved from the system resulting from increased agitation and subsequent desorption of the inhibitor from the pigment surface.

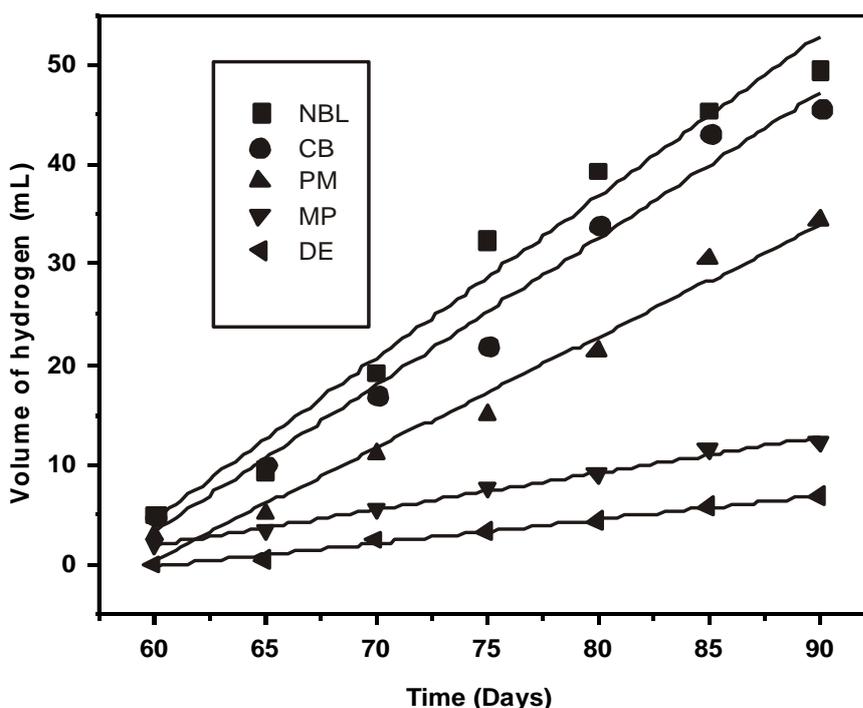
**Table 3: Inhibition efficiencies of CC, BC, PM, MP, and DE as a function of volume of H<sub>2</sub> gas evolved during corrosion of aluminium pigment powder at 40°C and pH 10.5**

Vol.H <sub>2</sub>	Inhibition efficiency (IE%)				
	NBL	CB	PM	MP	DE
10	95.4	96.5	98.0	98.5	99.0
20	94.8	96.0	97.7	98.4	98.7
30	94.1	95.4	97.3	98.1	98.5
40	93.7	95.0	97.1	97.9	98.3
50	93.2	94.6	96.9	97.7	98.2
50	92.8	94.2	96.6	97.6	98.1
70	92.6	94.0	96.5	97.5	98.0
80	92.2	93.6	96.3	97.4	97.9
90	91.7	93.3	96.1	97.2	97.8
100	91.6	93.1	96.0	97.1	97.7

### 3.4 Ninety (90) days inhibition experiment at 30°C

As a result of the slow reaction rate of the aluminium pigments in the presence of the plant extracts at 30°C /pH 10.5, the test at this temperature was allowed to run for 90 days. During the test period aluminium pigment dispersion was hand shaken every two days. The practical utility of this procedure is that there should be no formation of hydrogen or other chemical reaction during storage of the pigment product (paint etc) at room temperature, or at elevated temperature of 30-40°C [Muller, 2001a]. The uninhibited aluminium pigment dispersion reacted completely within one week. In contrast, there was no gas evolution from the dispersions containing the plant extracts for more than 60 days. The gas volumes recorded were obtained after 60 days of the test period.

Figure 5 shows the gas-volumetric result obtained at 30°C/pH 10.5 with addition of 5.0% v/v the various plant extracts (for clarity only the hydrogen volumes after 60 days are plotted). Linear variation for the volume of the hydrogen gas evolved with time was observed. The corrosion rate, R, which is obtained from the slope of the linear part of the gasometry plot for the test solutions are shown in Table 4.



**Figure 5: Variation of the volume of hydrogen evolved with time for aluminium pigment powder in 0.5 M NaOH in presence of 5.0% (v/v) plant extracts**

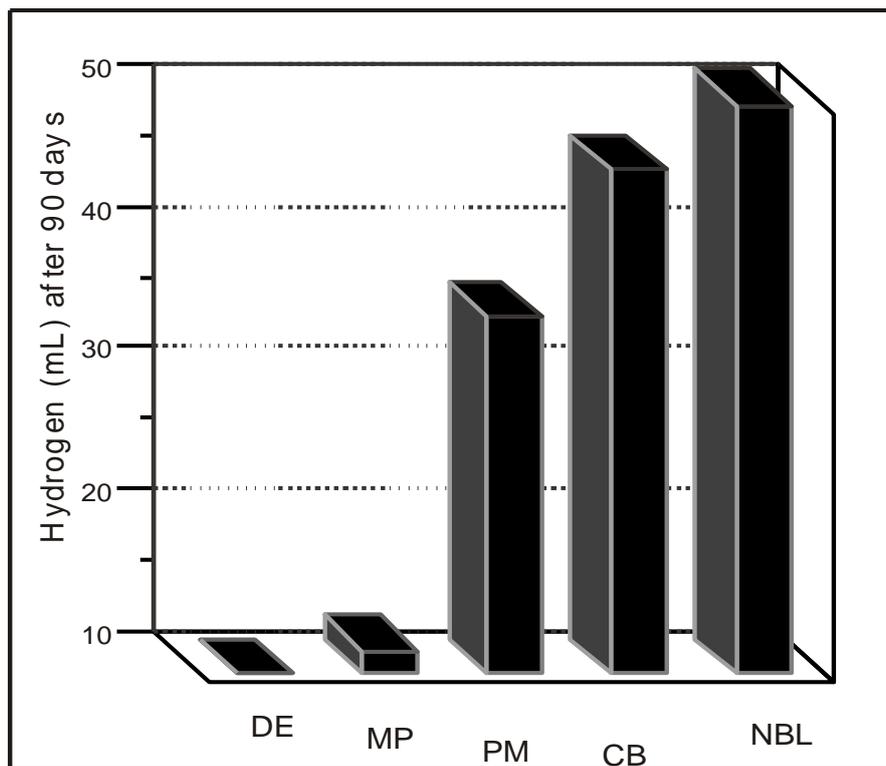
**Table 4:** The corrosion rate, obtained from gasometry plots in 0.5 M NaOH in presence of 5.0% (v/v) NBL, CB, PM, MP and DE plant extracts at 30°C and pH 10.5.

System	R (mLday <sup>-1</sup> cm <sup>-2</sup> )
Al dispersion +NBL	1.61
Al dispersion + CB	1.46
Al dispersion + PM	1.11
Al dispersion + MP	0.36
Al dispersion + DE	0.23

**Table 4** compares the corrosion rate of the of the aluminium dispersion in presence of the different

plant extracts. The corrosion rate of the uninhibited aluminium pigment was so fast that it reacted completely within one week hence it was not determined.

With regard to the practical applications of aluminium pigments in waterborne paints the hydrogen volume evolved after a certain period of time is the most important measured quantity which is then used to gauge the corrosion rate in the system. Figure 6 shows the total volume of hydrogen gas evolved in the presence the extracts within 90 days with the least volume coming from the dispersion containing DE and MP.



Plant extract

**Figure 6:** Comparison of the volumes of hydrogen evolved within 90 days from dispersion of aluminium pigment at pH 10.5 with addition of 5.0% (v/v) concentration of the plant extracts at 30°C

**Conclusion**

Aluminium pigments corrode in aqueous alkaline media with evolution of hydrogen which can be inhibited by the addition of inhibitors

The combination of the aluminium pigment with the plant extracts of NBL, CB, PM, MP, and DE significantly inhibited the corrosion process of aluminium pigment leading to an extended latency periods compared to that of the uninhibited aluminium pigment.

The latency period was found to depend on pH, temperature, and plant type and the duration of the latency showed the following order: DE>MP>PM>CB>NBL.

A linear correlation was obtained between inhibition efficiency and duration of latency for each extract.

Plant extracts can serve as effective, non toxic and cheap corrosion inhibitors for aluminium pigments in alkaline medium with DE as the most effective.

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