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Steel in Aqueous Solutions

Properties of Hybrid Composites

Highlights

Absorption Packed Column

Modeling and Output Feedback

Discovering Thoughts, Inventing Future

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Modeling and Output Feedback Distributed Control for an Absorption Packed Column

By M. Selatnia, R. Illoul & M.S. Boucherit
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Sammary- This work consists of modeling, simulation and multiple models control of an industrial absorption packed column designated to remove CO_2, from natural gas. The multiple models approach is an elegant way of turning nonlinear problems into linear ones. In this paper, we used the output feedback distributed control (ODC) coupled with local linearization of the model of the absorption packed column. We compared the results with those obtained with the traditional PID control and the results were satisfactory.

Keywords: multiple models, PDC control, methyldiethanolamine (MDEA) absorption packed column, LMI, lyapunov function.

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Modeling and Output Feedback Distributed Control for an Absorption Packed Column

M. Selatnia [α], R. Illoul [σ] & M.S. Boucherit [ρ]

Summary- This work consists of modeling, simulation and multiple models control of an industrial absorption packed column designated to remove CO_2, from natural gas. The multiple models approach is an elegant way of turning nonlinear problems into linear ones. In this paper, we used the output feedback distributed control (ODC) coupled with local linearization of the model of the absorption packed column. We compared the results with those obtained with the traditional PID control and the results were satisfactory.

Keywords: multiple models, PDC control, methyldiethanolamine (MDEA) absorption packed column, LMI, lyapunov function.

I. Introduction

The absorption packed column is a physicochemical separation unit largely used in the chemistry industry. It consists of a tube where we send gas mixtures in order to separate one or more compounds from the principal mixture. It is largely used for the separation of acid gases (CO_2, H_2S) from natural gas.

The model presented in this paper is a dynamic model of the absorption packed column and consists of a set of non linear partial differential equations; it is elaborated starting from considerations on CO_2 and MDEA mass balance in gas and liquid phases and considers also the energy balance [2]. We finally obtain a non linear distributed parameters system.

Few studies were carried out on modelling and controlling the absorption column. Crosby and Durbin [3] studied the performance of a state controller. Roffel [4] developed a sub-optimal output controller with state inequality constraint. Darwish and Fantin [5] used a decentralized control with pole placement. Petrovsky [6] developed a multivariable PI regulator. Najim [7] developed a self-adjusting regulator in the case of CO_2 absorption by a diethanolamine solution and also multilevel learning control [8]. It took again the problem later on with predictive control [9].

Few studies have also been published concerning the modelling and simulation of CO_2 absorption by aqueous solutions of MEA or MDEA on pilot and industrial columns [10-12].

For the model developed in our study, it seemed interesting for us to use the multiple models approach for the control of the absorption packed column because it enables us to obtain good performances for complex dynamics processes. We develop in first stage the PID regulation to compare the performances of the classical techniques with the performances of the Takagi-Sugeno multiple model approach.

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II. Modeling and Open Loop Simulation of the Industrial Absorption Packed Column

The absorption packed column presented here is located at Khrechba and is part of the In Salah Gaz project [17], it removes CO_2 from natural gas by using an aqueous solution of methyldiethanolamine (MDEA) as a washing liquid, It is a packed type column measuring 8 meter height and 4 meter in diameter with Pall rings to improve the surface of contact between phases. For a better elimination of CO_2 from the natural gas, the liquid flow (water+ MDEA) is counter-current with gas flow. The working pressure and temperature are respectively 71.5 bar at 55°C [2].

At contact between liquid and gas phase occurs on the surface of the Pall rings, CO_2 passes from the gas phase to the liquid phase; this diffusion is accelerated by chemical reaction of CO_2 with the MDEA in the liquid phase. The liquid flow (water+ MDEA) and the CO_2 concentration in the gas mixture are respectively selected as control variable and output variable.

Figure 1 : The absorption packed column of In Salah Gas (ISG)

a) Model Equations

In order to simplify the model, the following assumptions are done [2,13]:

- There is no resistance in gas phase
- The reaction between CO_2 and MDEA is fast (Ha>5)
- Axial dispersion is negligible in the gas phase and the liquid phase
- The MDEA does not pass in gas phase

The mass balance on an elementary section dz of the column for CO_2 in the gas phase is written [2,13,14]:

Quantity of aqueous solution at input Z = quantity of aqueous solution at the output (z+dz) + quantity of aqueous solution transferred from the liquid phase to the gas phase + accumulation.

Which gives:

$$(GC_{Ag})_z = (GC_{Ag})_{z+dz} + \varphi S\,dz + S\frac{dC_{Ag}}{dt}dz \qquad (1)$$

Where G (m3/s) is the volumetric gas flow, φ the CO_2 flow transferred from the gas phase to the liquid phase, S the section of the column and C_{Ag} (mol/m^3) the CO_2 concentration in the gas phase. Given U_G=G/S (m/s) the gas flow velocity, we obtain then:

$$U_g\frac{dC_{Ag}}{dz} + \varphi = -\frac{dC_{Ag}}{dt} \qquad (2)$$

The chemical reaction between CO2 and the MDEA is [10-12]:

$$CO_2 + R_1R_2NCH_3 + H_2O \rightarrow R_1R_2NCH_4^{+} + HCO_3^{-} \qquad (3)$$

The reaction rate rA has the following form [14,15]:

$$r_A = k\,C_{AL}\,C_{BL} \qquad (4)$$

Where k is the constant for reaction rate [14,15]:

$$k = 2{,}9610^5\exp\left(-\frac{5332.8}{T}\right) \qquad (5)$$

CAL is the CO_2 concentration in the liquid phase and CBL the MDEA concentration in the liquid phase. The mass balance for CO_2 in the liquid phase gives finally:

$$\varphi = [kC_{AL}C_{BL}] \qquad (6)$$

Which means that the totality of CO_2 transferred to the liquid phase reacts with the MDEA.

The mass balance for the MDEA in the liquid phase gives:

$$(LC_{Bl})_z = (LC_{Bl})_{z+dz} - [k\,C_{Al}\,C_{Bl}]\,S\,dz - S\frac{dC_{Bl}}{dt}dz \qquad (7)$$

Where L is the volumetric liquid flow. By taking account of (5) and noting by UL = L/S (m/s) the mean liquid flow velocity, we obtain:

$$U_l\frac{dC_{Bl}}{dz} - \varphi = \frac{dC_{Bl}}{dt} \qquad (8)$$

Our absorption packed column is finally described by the following set of partial derivative equations:

$$\begin{cases} U_g\dfrac{\partial C_{Ag}}{\partial z} + \varphi = -\dfrac{\partial C_{Ag}}{\partial t} \\ U_L\dfrac{\partial C_{BL}}{\partial z} - \varphi = \dfrac{\partial C_{BL}}{\partial t} \end{cases} \qquad (9)$$

The procedure to compute flow φ is given in [2] according to [14-16].

We have finally to consider the boundary conditions which for gas phase are the CO_2 concentration at the column bottom or input concentration CAGe and for liquid phase the MDEA concentration at the column top or input concentration CBLe.

$$\begin{cases} C_{Ag}\big|_{z=0} = C_{Age} \\ C_{BL}\big|_{z=h} = C_{BLe} \end{cases} \qquad (10)$$

Chemical reactions within the industrial column induces a strong heat emission and the appearance of a temperature gradient throughout the column; the temperature variation is approximately 5°C between the input and the output of the column, which leads us to establish an energy balance in order to describe the temperature changes which affects the various concentrations along the column [16]:

$$\begin{cases} U_g\dfrac{\partial T_g}{\partial z} + \dfrac{a.h_{g|l}\left(T_l - T_g\right)}{\left[\sum_i cp^g{}_i C^g{}_i\right]} = \dfrac{\partial T_g}{\partial t} \\ -U_L\dfrac{\partial T_l}{\partial z} + \dfrac{1}{\sum_i cp^l{}_i C^l{}_i}\left[\Delta H_r r_A - a.h_{g|l}\left(T_l - T_g\right)\right] = \dfrac{\partial T_l}{\partial t} \end{cases} \qquad (11)$$

With:

C_i^g: concentration in gas phase at the interface (mol/m^3)

C_i^l : concentration in liquid phase at the interface (mol/m^3)

cp_i^g : Specific heat in the gas phase at the interface (J/mol.K)

$h_{g/l}$: coefficient of heat transfer (convection) (J/m2.K.s)

T_i: Liquid temperature (K)

Tg: Gas temperature (K)

ΔHr: enthalpy of the reaction (J/mol)

cp_i^l: specific heat in the liquid phase at the interface (J/mol.K)

We finally take into account the boundary conditions for the temperature which are the temperatures for gas and the liquid at the column input.

$$\begin{cases} T_g\big|_{z=0} = T_{ge}\ , \dfrac{\partial T_l}{\partial z}\Big|_{z=0} = 0 \\ T_l\big|_{z=h} = T_{le}\ , \dfrac{\partial T_g}{\partial z}\Big|_{z=h} = 0 \end{cases} \qquad (12)$$

b) Model validation

A test was carried out on our industrial absorption column to compare the output CO_2 concentration given by the model with the real one and this for a step input variation of 10 t/h. the data were collected on a horizon of 6800 seconds. The results are grouped in figure 2 where we represent respectively, the flows of MDEA and gas at the output and then the concentrations of CO_2 at the column output either experimental or given by the model [2].

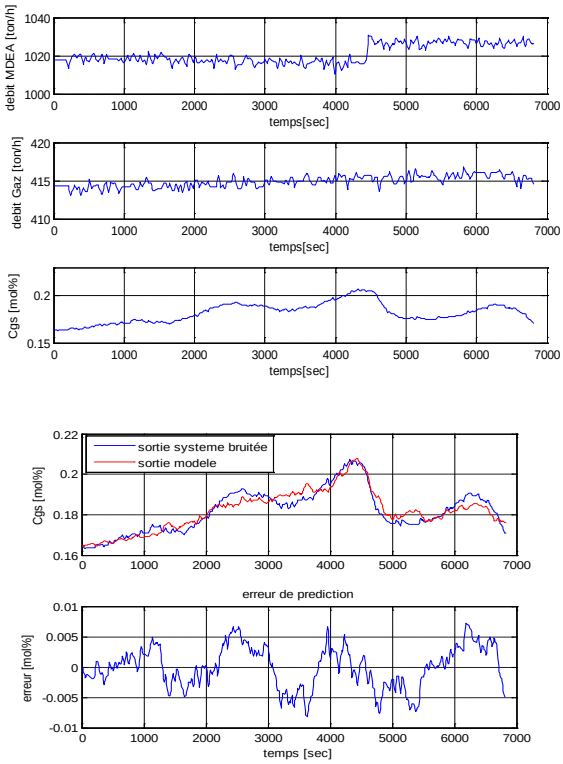

Figure 2 : Output CO_2 Concentrations from the system and the model

We note that the model dynamics of the CO_2 concentration at the column output agree with the experimental results.

c) Open loop Simulation of the industrial column

By considering the equations (2) and (8), the dynamic model of the absorption column is that of a nonlinear, distributed parameters system. The results from open loop simulations are presented in figures 3 and 4.

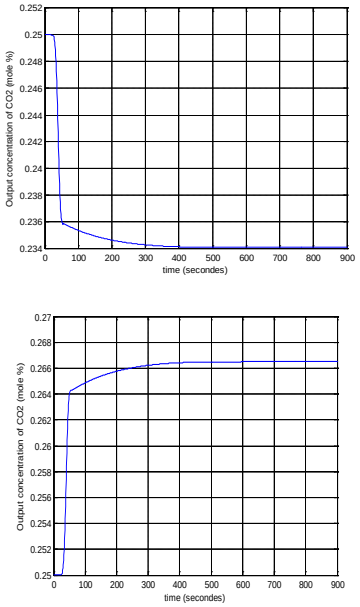

Figure 3 : Output CO_2 Concentration for an input step disturbance of ±5 % on gas concentration

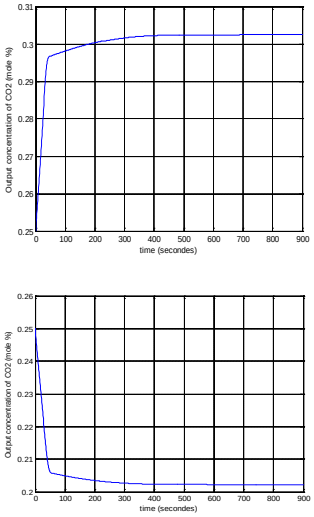

Figure 4 : Output CO_2 Concentration for an input step disturbance of ±10 % on gas flow

Simulations show that the system is stable. It presents a dead time in response to a step input disturbance on the CO_2 concentration due to the gas propagation along the absorption column.

III. PID Regulation of the Industrial Absorption Packed Column

We apply a PID regulation to the dynamic model of our absorption packed column. We choose a sampled control with a sampling period of 10 seconds. The reference for the input CO_2 concentration is 0.25mole %, which corresponds to a concentration of 7.05 mole CO_2/m^3 .The parameters of the PID regulator were optimized using trial and error.

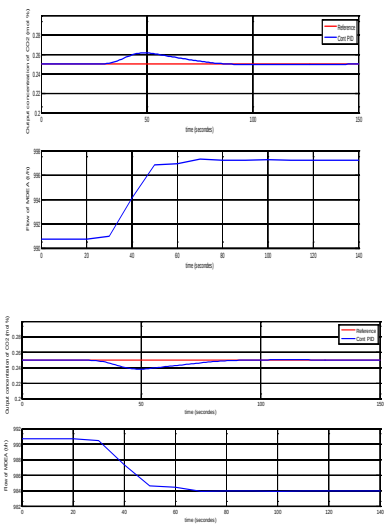

Figure. 5 : Output CO_2 Concentration and washing liquid flow for an input step disturbance of ±5 % on gas concentration

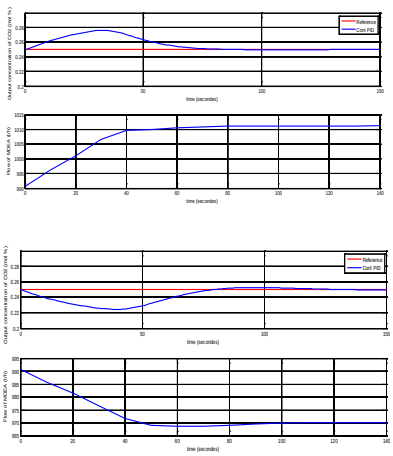

Figure 6 : Output CO_2 Concentration and washing liquid flow for an input step disturbance of ± 10 % on gas flow

The simulation results are satisfactory; the PID regulator cancels the permanent error and ensures a quick response due to the derivative action. The regulation shows a net asymmetrical behaviour between responses to positive and negative step input disturbances due to the strong non linearity of the relationship between the input and the output

IV. Multiple Models Control of the Industrial Absorption Packed Column

a) Introduction

The multiple models is a non linear modeling technique which allows to achieve a good compromise between precision and model complexity. In the light of the numerous work related to it in recent years [18], [19], [20], it arises a great interest, especially in applications dealing with simulation and control. It can also be seen as a particular fuzzy modeling technique [21], [22], corresponding to a Takagi Sugeno (TS) approach [23]. A TS model is a composed of a finite number of linear models connected with non linear functions called membership functions and verifying the convex mapping property (i.e. they are non negative and their sum is equal to 1). It allows us to solve various problems of control, observation and diagnosis for non linear systems with linear techniques.

The approach associated with multiple models in control is known as the Parallel Distributed Compensation (PDC) [24]. This method is based on a set of linear controllers designed for each linear model, and stability of the overall closed loop is guaranteed via a Lyapunov function common to all the linear models.

In this paper, we identify the absorption packed column as a multiple model of the TS type and proposes a control based on Output feedback Distributed Control (ODC).

b) Problem formulation

i. The Multiple model approach

The multiple models have three basic structures: Coupled states (TS), uncoupled states [25] and hierarchical structure. The coupled states Structure (TS) is the most popular in the analysis and synthesis of the multiple models. It is written in the following form:

$$\begin{cases} \dot{x}(t) = \sum_{i=1}^{M} \mu_i(z(t)) \, (A_i x(t) + B_i u(t)) \\ y(t) = \sum_{i=1}^{M} \mu_i(z(t)) \, C_i x(t) \end{cases} \quad (13)$$

$x(t) \in R^n$ being the state vector, $u(t) \in R^m$ the input vector, $y(t) \in R^p$ the output vector $z(t) \in$ R is the decision variable or premises and the matrices $A_i \in R^{n \times n}$, $B_i \in R^{n \times m}$, $et\ C_i \in R^{p \times n}\ \forall\ i = 1, \ldots\ldots, M$ are constant and supposed to be known.

The activation function or membership function $\mu_i(z(t)$ determines the degree of activation of the i[th] local model. It allows a progressive passage from this model to the other close local models. These functions can depend of the measurable variables of the system (the input and output signals) or of the non measurable variables of the system (the states). They can be of triangular or Gaussian form and satisfy the properties of convex mapping:

$$\begin{cases} \dot{x}(t) = \sum_{i=1}^{M} \mu_i(z(t)) \, (A_i x(t) + B_i u(t)) \\ y(t) = \sum_{i=1}^{M} \mu_i(z(t)) \, C_i x(t) \end{cases} \quad (14)$$

The multiple models can be viewed as universal approximators since any nonlinear system can be approximated by a multiple models representation with sufficient accuracy and this simply by increasing the number of sub-models. In practice, a reduced number of sub-models can be sufficient to obtain a satisfactory

approximation, and we can use the tools of linear systems analysis to achieve this goal.

There are three approaches largely used in the literature allowing us to obtain a TS model: Identification, transformation by nonlinear sectors [26], or linearization. This last one is used in this work (figure 7).

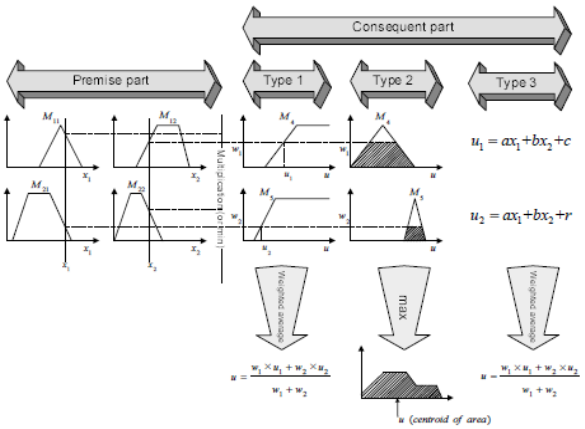

Figure 7 : PDC control for TS multiple models

Using convex analysis for regulator synthesis, the multiple models allows us to obtain control laws by the simultaneous resolution of a finite number of Linear Matrix Inequalities (LMI). In this case, the number of LMI inequalities is polynomial with respect to the number of local models. Thus, it is advisable to minimize the number of local models to limit the conservatism of the method.

In the case of TS multiple models, this technique of regulator synthesis corresponds to the PDC method, It supposes that all the linear sub-models are at least stabilizable. Subsequently, they will also be supposed controlled.

Given the TS model given by equation (13), a control law resulting from PDC synthesis will thus be the combination of linear control laws for each sub-model, given by:

$$u(t) = -\sum_{i=1}^{M} \mu_i(z(t)) F_i x(t) \qquad (14)$$

Applying this control law to the TS multiple model, we obtain in closed loop:

$$\begin{cases} \dot{x}(t) = (A_z - B_z F_z)x(t) \\ y(t) = C_z x(t) \end{cases} \qquad (15)$$

Or, in a more explicit way:

$$\{\dot{x}(t) = \sum_{i=1}^{M}\sum_{j=1}^{M} \mu_i(z(t))\,\mu_j(z(t))(A_i - B_i F_j)x(t) \quad (16)$$

The stability conditions for the closed loop system amounts to find a control gain Fj such that the derivative of the candidate Lyapunov function associated with the system is negative. Stabilizing the system thus amounts to solve the following problem:

Find a positive definite matrix P and Fi matrices, i=1. , M such that:

$$(A_z - B_z F_z)^T P + P(A_z - B_z F_z) < 0 \qquad (17)$$

We notice that this inequality is non linear with respect to P and Fi. By using the congruence of the symmetrical full row matrix:

$$X = P^{-1} \qquad (18)$$

We get:

$$XA^T{}_z + A_z X - XF_z{}^T B^T{}_z - B_z F_z X < 0 \qquad (19)$$

By using the bijective variable change

$$M_i = F_i X, i = 1, \ldots, M, \qquad (20)$$

The problem becomes LMI in variables X and Mi.

$$\gamma_{ij} = XA^T{}_i - M^T{}_j B^T{}_i + A_i X - B_i M_j < 0 \qquad (21)$$

We finally get:

$$\sum_{i=1}^{M}\sum_{j=1}^{M} \mu_i(z(t))\mu_j(z(t))\gamma_{ij} \qquad (22)$$

And we can express the following result:

Theorem 4.1 [24] Given a continuous TS model, the PDC control law (14) and the γij, if it exists a positive definite matrix X and Mi matrices, such that (21) is satisfied for all i,j=1,…, M, then the closed loop is overall asymptotically stable. Moreover, if the problem has a solution, the gains of the PDC control are given by:

$$F_i = M_i X^{-1} \qquad (23)$$

And the PDC control is:

$$u(t) = -\sum_{i=1}^{M} \mu_i(z(t)) F_i x(t) \qquad (24)$$

If $F_i = F, \forall i = 1, \ldots, M$, then we define a linear control law. In practice, to determine the matrix P and the control gain F_i, we have to solve (21) for all *i,j*=1,…,M. In the particular case where the multiple models verify the positive co linearity of the input matrices, thais:

$$B_i = B, \forall i \in I_n \qquad (25)$$

The closed loop multiple models system of (16) is rewritten without the crossing terms B_iF_j:

$$\dot{x}(t) = \sum_{i=1}^{M} \mu_i(z(t))(A_i - B_i F_i)x(t) \qquad (26)$$

The stability conditions of theorem 4.1 reduce then to the stability of the dominant models: P>0 ,

$$(A_i - B_i K_i)P + P(A_i - B_i K_i) < 0, \forall i = 1, \ldots, M \qquad (27)$$

Substituting Bi by B, the control law leads to similar conditions.

c) Multiple models identification

The structural identification of a multiple models representation consists in the determination of the local models structures and the operation zones (or validity zones) for each local model [25]. The local models can be of various structures but in general we use simple structures, such as linear models.

The identification leads to a family of functions parameterized by the parameters vector $\underline{\theta}_i$ defining the structure of the i[th] local model, and the parameters vector $\underline{\beta}_i$ characterizing the zone of validity of this local model. The parametric estimate consists in determining for each local model *i* the parameters vector:

$$\underline{\Theta}_i = \begin{bmatrix} \underline{\theta}_i^T & \underline{\beta}_i^T \end{bmatrix}^T$$

The parametric estimation (also called training) is done on the basis of minimization of a functional binding the inputs and outputs system to the characteristics parameters of the model.

$$J_G = \frac{1}{2}\sum_{k=1}^{N}(y_s(t) - \hat{y}(t))^2 = \frac{1}{2}\sum_{k=1}^{N}\left[y_s(t) - \sum_{i=1}^{M}\omega_i\left(\underline{\zeta}(t), \underline{\beta}_i\right) f_i\left(\underline{\varphi}(t), \underline{\theta}_i\right)\right]^2 \quad (28)$$

In order to simplify the model, we choose linear sub-models of ARX type (auto regressive with exogenous inputs) with 3 inputs (system MISO):

- Liquid Flow Ul [ton/h]
- Gaz flow Ug [ton/h]
- Input CO_2 concentration Cge [mol%]

The choosen local models are second order ones, they are written in the following form:

$$C_{gsi}(t+1) = A_{i1}C_{gs}(t) + A_{i2}C_{gs}(t-1) + B_iU_l(t) + C_iU_g(t) + D_iC_{ge}(t) + P_i + e_i(t+1) \quad (29)$$

With:

i= *1*,…. ,M : local model indices

$C_{gsi}(t+1)$:output (concentration of CO_2) of local model i

e_i (t+1) : Gaussian white noise

A_{i1} , A_{i2} , B_i, C_i, D_i, P_i : parameters of local model i

We choose Gaussian membership functions with the decision variable being the system output at time t.

The expression of the membership function is:

$$\omega_i\left(C_{gs}(t), [c_i, \sigma]\right) = \frac{\exp\left(-\frac{(C_{gs}(t)-c_i)^2}{2\sigma^2}\right)}{\sum_{i=1}^{M}\exp\left(-\frac{(C_{gs}(t)-c_i)^2}{2\sigma^2}\right)} \quad (30)$$

Where c_i is the mean. and σ the standard deviation for the Gaussian membership function

The global identification diagram is:

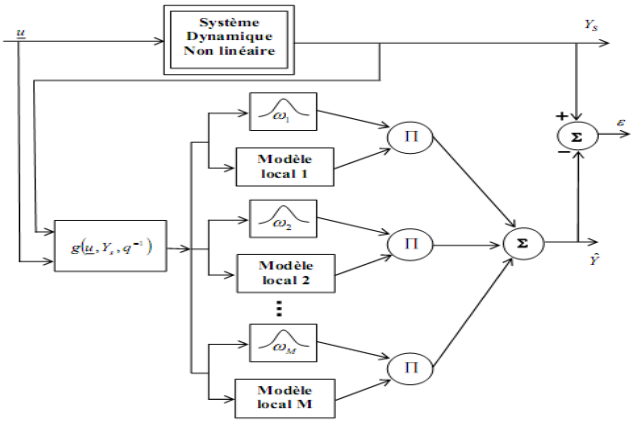

Figure 8 : Structure of the multiple models identification

Training data

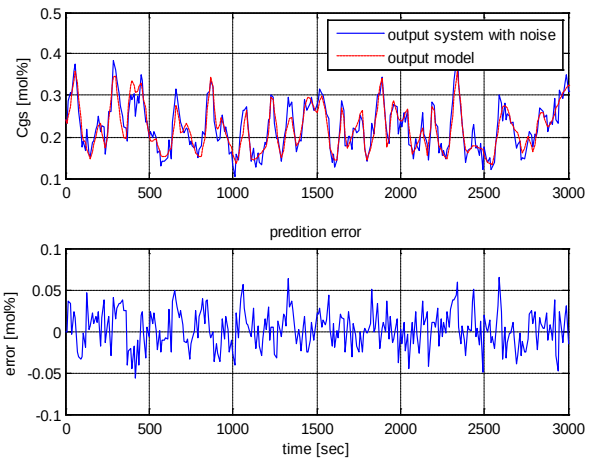

Figure 9 : Training with the ARX structure

Validating data

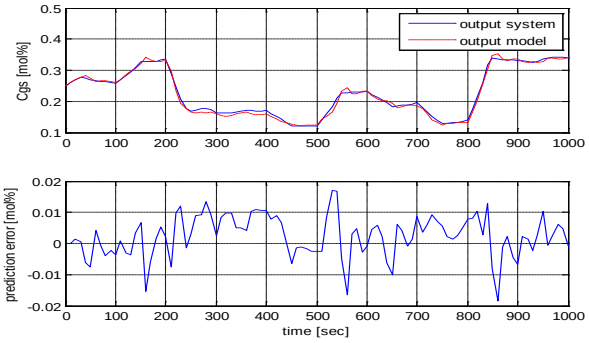

Figure 10 : Validating with the ARX structure

d) Implementation of the method and simulation results

The decision variable z(t) is in our case the state vector x(t) output gas concentration of the industrial column C_{gs}, $x(t) = [C_{gs}(t)\ C_{gs}(t-1)]^T$. A multiple models with four sub-models can easily be obtained in the form:

$$\dot{x}(t) = \sum_{i=1}^{4} \mu_i\ (x(t))(A_i - B_i F_i)x(t) \qquad (31)$$

With the memberships functions $\mu_i(x(t))$ either triangular or Gaussian. The parameters of the four models are:

$$A_1 = \begin{pmatrix} -0.4436 \\ 0.5744 \end{pmatrix}\ A_2 = \begin{pmatrix} -0.4772 \\ -0.2732 \end{pmatrix}\ A_3 = \begin{pmatrix} -1.8937 \\ 0.3093 \end{pmatrix}$$
$$A_4 = \begin{pmatrix} 0.5339 \\ -0.2304 \end{pmatrix} \qquad (32)$$
$$B_1 = (-0.0006)\ B_2 = (-0.0006)\ B_3 = (-0.0006)$$
$$B_4 = (-0.0006) C_1 = C_2 = C_3 = C_4 = \begin{pmatrix} 1 \\ 0 \end{pmatrix}$$

And the Output Feedback Distributed Control (ODC) is:

$$U_L(t) = -\sum_{i=1}^{4} \mu_i \left(C_{gs}(t)\right) F_i x(t) + U_{Lin} \qquad (33)$$

U_L is the liquid flow velocity, and U_{Lin} the mean flow velocity corresponding to the choosen operating point of the column.

The F_i feedback gains are determined by resolution of LMI in order to ensure good regulation performances, less than a 0.25% variation of CO_2 around the operating points.

The selected gain is finally $F_i = [980\ \ 985\ \ 995\ 1002]$ t/h

i. *Closed loop simulation*

Case (a): triangular membership functions

The column simulation shows us the output CO_2 concentration for step input disturbances on either the gas flow or the CO_2 concentration. The evolutions of the output CO_2 concentration and the corresponding control are on figures 11,12 and 13.

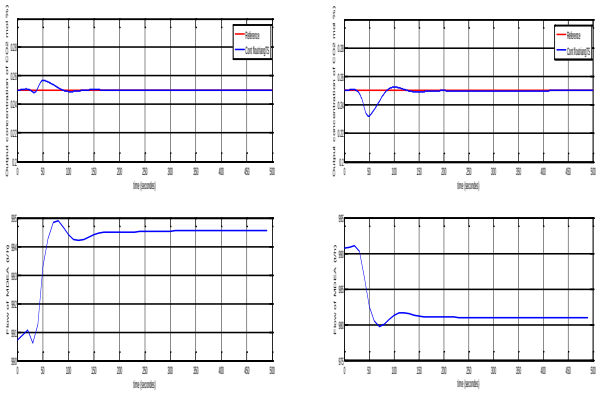

Figure 11 : Output CO_2 Concentration and washing liquid flow for a step input disturbance of ± 5 % on gas concentration

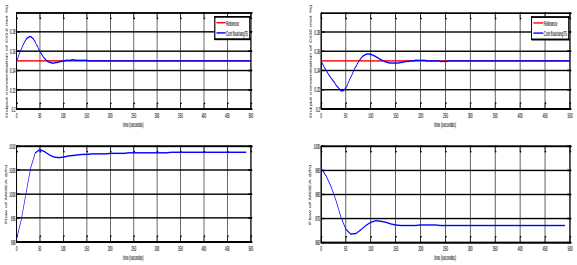

Figure 12 : Output CO_2 Concentration and washing liquid flow for a step input disturbance of ± 10 % on gas flow

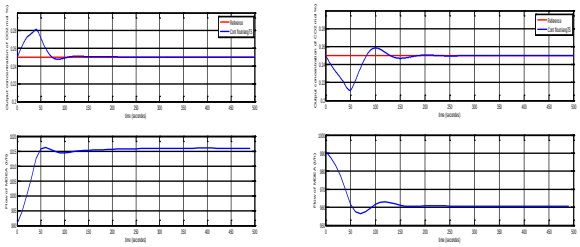

Figure 13 : Output CO_2 Concentration and washing liquid flow for a step input disturbance of ± 5 % on gas concentration and ± 10 % on gas flow

Case (b): Gaussian membership functions

The evolutions of the output CO_2 concentration and the corresponding control are on figures 14, 15 and 16.

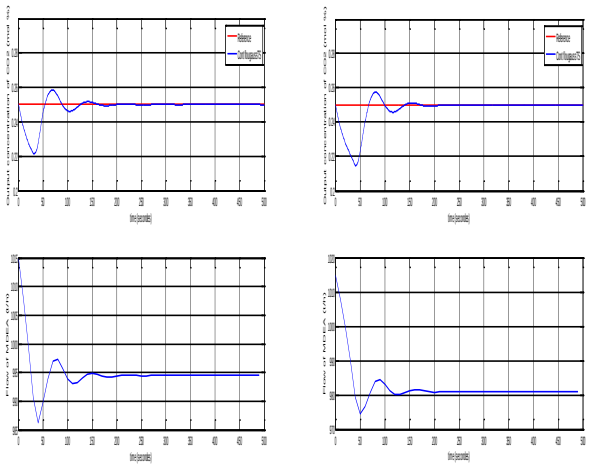

Figure 14 : Output CO_2 Concentration and washing liquid flow for a step input disturbance of ± 5 % on gas concentration

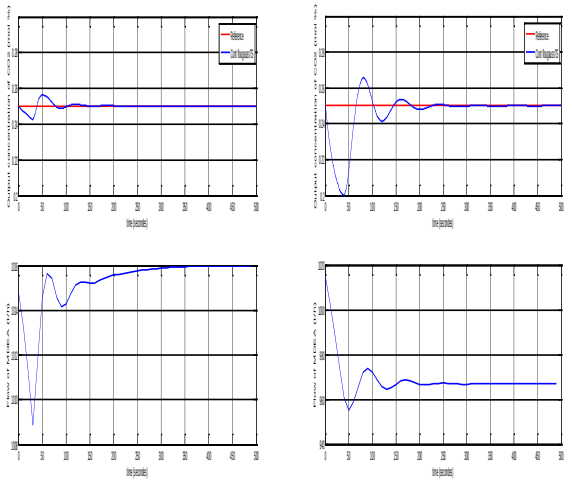

Figure 15 : Output CO_2 Concentration and washing liquid flow for a step input disturbance of ± 10 % on gas flow

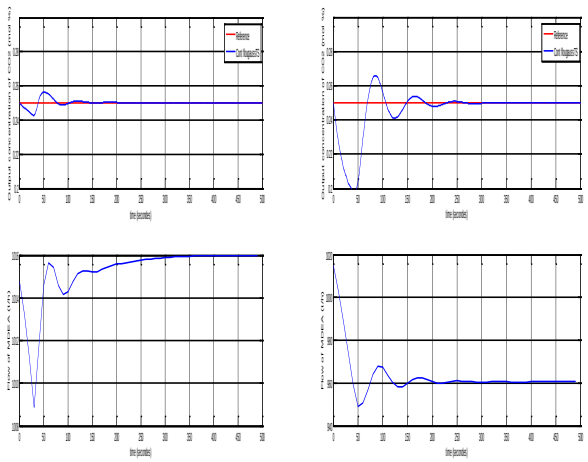

Figure 16 : Output CO_2 Concentration and washing liquid flow for a step input disturbance of ± 5 % on gas concentration and ± 10 % on gas flow

V. Discussions

The oscillations of the output CO_2 concentration for the industrial column are mainly due to fuzzy control, but the operating point of is quickly reached in less than 50 seconds . We note on all the curves (5, 6 and 11-16) that disturbance is always rejected with both PID and ODC regulation. But comparison of the peak values as well as the oscillations show us that ODC control acts more quickly than PID control.

VI. Conclusion

In this paper, a TS multiple models obtained by linearization was used for the regulation of an industrial absorption packed column used for gas washing. We take a reduced number of submodels, four, to ease identification. The results obtained with output distributed feedback (ODC) are better than those by classical PID regulation, for a method which is not significantly complicated. Further investigations will be undertaken in the use of multiple models in control, multiple observers and multiple models in systems diagnosis.

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Corrosion Inhibition of Mild Steel in Aqueous Solutions using Nonionic Surfactants

By A. S. Fouda, A. M. Attia & A. M. Rashed
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I. Introduction

Corrosion is a fundamental process playing an important role in economics and safety, particularly for metals. The use of inhibitors is one of the most practical methods for protection against corrosion, especially in acidic media [1]. Most Well-known acid inhibitors are organic compounds containing nitrogen , sulfur, and oxygen atoms. Among them, surfactant inhibitors have many advantages such as high inhibition efficiency, low price, low toxicity, and easy production [2-6]. Ionic surfactants have been used for the corrosion inhibition of [iron] [7-12], copper [13], aluminum [14-16], and other metals [17, 18] in different corroding media. The adsorption of the surfactant on the metal surface can markedly change the corrosion-resisting property of the metal [19, 20] and so the study of the relations between the adsorption and corrosion inhibition is of great important. Nonionic surfactants have shown a high inhibition efficiency for iron in both HC [l21, 22] and H_2SO_4 [23] solutions. Nonionic surfactants were studied as corrosion inhibitors for different metals and in different media by several authors [24-28].

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As a nonionic surfactant, Tween 20 and 60 have rarely been studied as inhibitors for mild steel in HCl. For this reason, the objective of the present work is to investigate the inhibition action of Tween 20 and 60 in 0.5 M HCl at 25-55 ^{o}C using chemical and electrochemical techniques.

II. Experimental Methods

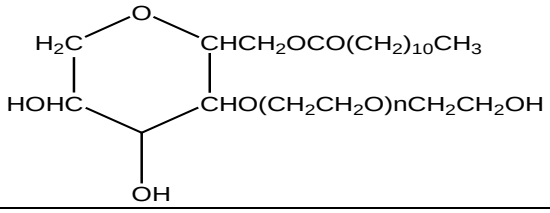
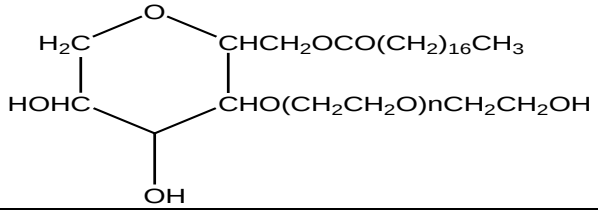
a) Materials

Tests were performed on mild steel of the following composition (weight %):0.15-0.20 % C, 0.60-0.90 % Mn, 0.04 % P, 0.05 % S, and the remainder Fe

b) Inhibitors

Tween 20 and 60 obtained from Shanghai Chemical Reagent Company of China and used as received. Table 1 shows the molecular structure of the Tweens. It is obvious that Tweens are O-heterocyclic compounds. The main functional group is hydroxyl. The molecular weights of Tweens are also high because of a number of units CH_2CH_2O.

Table 1 : The names and molecular structures of the investigated Tweens

Cpd. No.	Name	Structure
1	Tween 20	
2	Tween 60	

c) Solutions

The aggressive solutions, 0.5 M HCl were prepared by dilution of analytical grade HCl (37%) with bi-distilled water. The concentration range of the inhibitors used was 20-120 ppm

d) Gravimetric measurements

Seven parallel mild steel sheets of 2.5 × 2.0 × 0.06 cm were abraded with emery paper (grade 320–500–800) and then washed with bidistilled water and acetone. After accurate weighing, the specimens were immersed in a 250 ml beaker, which contained 250 ml of HCl with and without addition of different concentrations of Tween-20 and 60.

All the aggressive acid solutions were open to air. After 3 h, the specimens were taken out, washed, dried, and weighed accurately. The average weight loss of seven parallel mild steel sheets could be obtained. The inhibition efficiency (%IE) and the degree of surface coverage ,θ, of Tween-20 and 60 for the corrosion of mild steel was calculated as follows [29],

$$\%IE = \theta \times 100 = [W^o - (W/W^o)] \times 100 \qquad (1)$$

Where W^o and W are the values of the average weight loss without and with addition of the inhibitor, respectively.

e) Polarization measurements

Polarization experiments were carried out in a conventional three-electrode cell with a platinum counter electrode and a saturated calomel electrode (SCE) coupled to a fine Luggin capillary as the reference electrode. The working electrode was in the form of a square cut from mild steel embedded in epoxy resin of polytetrafluoroethylene (PTFE) so that the flat surface was the only surface in the electrode. The working surface area was 1.0×1.0 cm. Tafel polarization curves were obtained by changing the electrode potential automatically from -600 to +300 mV at open circuit potential with a scan rate 5 mVs^{-1}. Stern-Geary method [30] used for the determination of corrosion current is performed by extrapolation of anodic and cathodic Tafel lines to a point which gives log i_{corr} and the corresponding corrosion potential (E_{corr}) for inhibitor free acid and for each concentration of inhibitor. Then i_{corr} was used for calculation of inhibition efficiency and surface coverage (θ) as below:

$$\%\ IE = \theta \times 100 = [1- (i_{corr(inh)} / i_{corr(free)})] \times 100 \qquad (2)$$

Where $i_{corr(free)}$ and $i_{corr(inh)}$ are the corrosion current densities in the absence and presence of inhibitor, respectively.

Impedance measurements were carried out in frequency range from 100 kHz to 10 mHz with amplitude of 5 mV peak-to-peak using ac signals at open circuit potential. The experimental impedance were analyzed and interpreted on the basis of the equivalent circuit. The main parameters deduced from the analysis of Nyquist diagram are the resistance of charge transfer R_{ct} (diameter of high frequency loop) and the capacity of double layer C_{dl} which is defined as:

$$C_{dl} = 1/ (2 \pi f_{max} R_{ct}) \qquad (3)$$

The inhibition efficiencies and the surface coverage (θ) obtained from the impedance measurements are defined by the following relation:

$$IE\ \% = \theta \times 100 = [1- (R^{\circ}_{ct}/R_{ct})] \times 100 \qquad (4)$$

Where R°_{ct} and R_{ct} are the charge transfer resistance in the absence and presence of inhibitor, respectively.

Electrochemical frequency modulation, EFM, was carried out using two frequencies 2 and 5 Hz. The base frequency was 0.1 Hz, so the waveform repeats

after 1 s. The higher frequency must be at least two times the lower one. The higher frequency must also be sufficiently slow that the charging of the double layer does not contribute to the current response often; 10 Hz is a reasonable limit. The Intermodulation spectra contain current responses assigned for harmonical and intermodulation current peaks. The larger peaks were used to calculate the corrosion current density (i_{corr}), the Tafel slopes (β_c and β_a) and the causality factors CF-2& CF-3[31].

The electrode potential was allowed to stabilize 30 min before starting the measurements. All the experiments were conducted at 30 ± 1°C. Measurements were performed using Gamry Instrument Potentiostat/Galvanostat/ZRA. This includes a Gamry framework system based on the ESA 400. Gamry applications include DC105 for corrosion measurements, EIS300 for electrochemical impedance spectroscopy and EFM 140 for electrochemical frequency modulation measurements along with a computer for collecting data. Echem Analyst 5.58 software was used for plotting, graphing, and fitting data.

III. Results and Discussion

a) Weight loss measurements

The weight loss-time curves of mild steel with the addition of tween 60 in 0.5 M HCl at various concentrations is shown in Fig. 1 as an example. The curves of Fig. 1 show that the weight loss values of mild steel in 0.5 M HCl solution containing Tween 60 decrease as the concentration of the inhibitor increases; i.e., the corrosion inhibition strengthens with the nonionic surfactant concentration. This trend may result from the fact that the adsorption of surfactant on the mild steel increases with the increase of inhibitor concentration thus the mild steel surface is efficiently separated from the medium by the formation of a film on its surface [32].

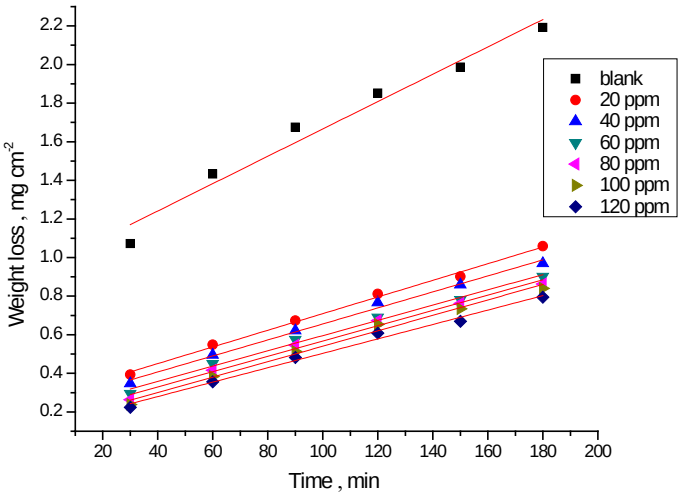


Figure 1 : Weight loss-time curves of mild steel in 0.5 M HCl in the absence and presence of different concentrations of tween 60 at 25°C

b) Potentiodynamic polarization measurements

Figure 2 shows the anodic and cathodic Tafel polarization curves for mild steel in 0.5 M HCl in the absence and presence of varying concentrations of tween 60 at 25°C as an example. The effect of temperature on the IE % for Tweens was studied using this technique. From Fig. 2, it is clear that both anodic metal dissolution and cathodic reduction reactions were inhibited when Tweens were added to 0.5 M HCl and this inhibition was more pronounced with increasing inhibitor concentration. Tafel lines are shifted to more negative and more positive potentials with respect to the blank curve by increasing the concentration of the Tweens. This behavior indicates that the undertaken additives act as mixed type inhibitors [33, 34]. The results show that the increase in inhibitor concentration leads to decrease the corrosion current density (i_{corr}), but the Tafel slopes (β_a, β_c),are approximately constant indicating that the retardation of the two reactions (cathodic hydrogen reduction and anodic metal dissolution) were affected without changing the dissolution mechanism [35-38] (Table 2).

Table 2 : The effect of inhibitor concentration on the free corrosion potential (E_{corr}), corrosion current density (i_{corr}), Tafel slope (β_a , β_c) , inhibition efficiency(IE%) , degree of surface coverage (Θ) ,corrosion rate (C.R) , for the corrosion of mild steel in 0.5 M HCl at 25°C

Comp.	Conc., ppm	- E_{corr} mV vs SCE	i_{corr}, $\mu A\ cm^{-2}$	$-\beta_c$, $mVdec^{-1}$	β_a, $mVdec^{-1}$	θ	IE%	C.R , $\mu m\ y^{-1}$
Blank	0.0	484	425.6	118	89	0.000	00.0	4940
Tween 20	20	486	162.2	104	79	0.619	61.9	1883
	40	486	157.9	106	82	0.629	63.0	1833
	60	477	141.0	103	73	0.669	66.9	1637
	80	478	136.0	106	80	0.680	68.0	1579
	100	485	115.2	97	75	0.729	72.9	1337
	120	474	103.7	105	70	0.756	75.6	1204
Tween 60	20	457	115.3	106	69	0.729	73.0	1339
	40	455	98.91	103	67	0.768	76.8	1148
	60	450	91.8	107	69	0.784	78.4	1066
	100	447	85.56	105	65	0.799	80.0	993
	120	452	84.12	104	69	0.802	80.2	976

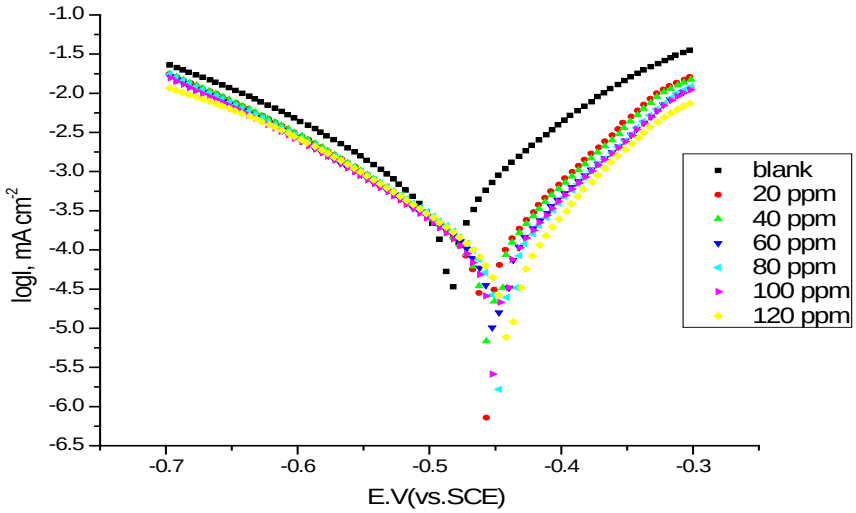


Figure 2 : Potentiodynamic polarization curves for corrosion of mild steel in 0.5 M HCl in the absence and presence of different concentrations of tween 60 at 25°C

c) Electrochemical impedance spectroscopy (EIS) measurements

The effect of inhibitor concentration on the impedance behavior of mild steel in 0.5 M HCl solution at 25°C is presented in Fig. 3a. The curves show a similar type of Nyquist plots for mild steel in the presence of various concentrations of Tween 60. Similar curves were obtained for Tween 20 (not shown). The existence of single semi-circle showed the single charge transfer process during dissolution which is unaffected by the presence of inhibitor molecules. Deviations from perfect circular shape are often referred to the frequency dispersion of interfacial impedance which arises due to surface roughness, impurities, dislocations, grain boundaries, adsorption of inhibitors, and formation of porous layers and in homogenates of the electrode surface [39, 40]. Inspections of the data reveal that each impedance diagram consists of a large capacitive loop with one capacitive time constant in the Bode–phase plots (Fig.3b). The electrical equivalent circuit model is shown in Fig. (4). It used to analyze the obtained impedance data. The model consists of the solution resistance (R_s), the charge-transfer resistance of the interfacial corrosion reaction (R_{ct}) and the double layer capacitance (C_{dl}). Excellent fit with this model was obtained with our experimental data. EIS data (Table 3) show that the R_{ct} values increases and the C_{dl} values decreases with increasing the inhibitor concentrations. This is due to the gradual replacement of water molecules by the adsorption of the inhibitor molecules on the metal surface, decreasing the extent of

dissolution reaction. The high (R_{ct}) values, are generally associated with slower corroding system [41, 42].

The decrease in the C_{dl} can result from the decrease of the local dielectric constant and/or from the increase of thickness of the electrical double layer suggested that the inhibitor molecules function by adsorption at the metal/solution interface [43]. The % IE obtained from EIS measurements are close to those deduced from polarization measurements. The order of inhibition efficiency obtained from EIS measurements is as follows: Tween 20 > Tween 60.

Table 3 : EIS data of mild steel in 0.5 M HCl and in the absence and presence of different concentrations of Tweens at 25°C

Comp.	Conc., ppm	R_{CT}, Ω cm^2	C_{dl}, μFcm^{-2}	θ	IE%
Blank	00	25.67	151.4	0.000	00.0
Tween 20	20	79.05	92.0	0.675	67.5
	40	82.47	83.1	0.689	68.9
	60	85.57	76.9	0.700	70.0
	80	104.9	74.6	0.755	75.5
	100	117.0	71.8	78.1	78.1
Tween 60	20	152.4	98.2	0.832	83.2
	40	193.9	90.3	0.868	86.8
	60	215.1	89.0	0.881	88.1
	80	233.4	84.9	0.890	89.0
	100	248.5	84.2	0.897	89.7

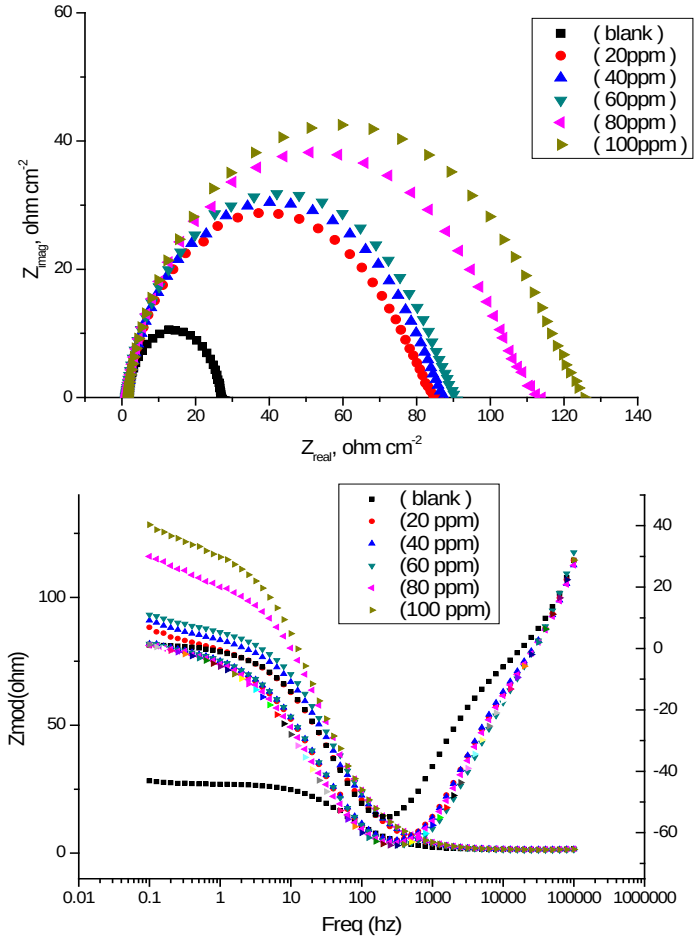


Figure 3 : Nyquist (a) and Bode (b) plots for mild steel in 0.5 M HCl in the absence and presence of tween 20 at 25°C

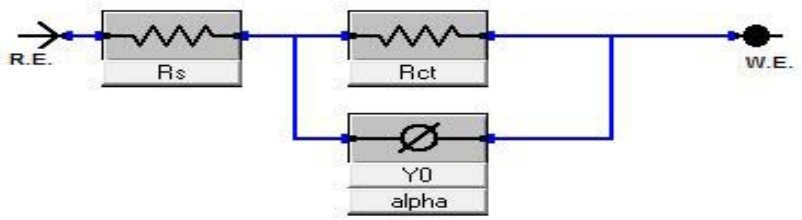


Figure 4 : Electrical equivalent circuit model used to fit the results of impedance

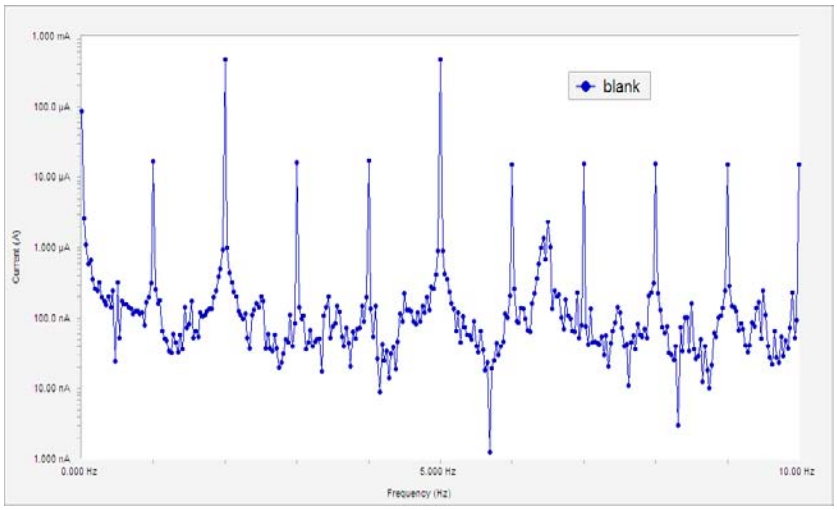
d) Electrochemical frequency modulation (EFM) measurements

The EFM is a nondestructive corrosion measurement technique that can directly give values of the corrosion current without prior knowledge of Tafel constants. Like EIS, it is a small ac signal. Intermodulation spectra obtained from EFM measurements are presented in Fig. (5 a, b) are examples of mild steel in 0.5 M HCl solutions devoid of and containing 100 ppm concentrations of Tween 20 at 25˚C. Similar intermodulation spectra were obtained for other tween (not shown). Each spectrum is a current response as a function of frequency. The calculated corrosion kinetic parameters at different concentrations of the investigated compounds in 0.5 M HCl at 25 °C (i_{corr}, β_a, β_c, CF-2, CF-3 and IE %) are given in Table (4).

From Table 4, the corrosion current densities decrease by increasing the concentration of investigated compounds and the inhibition efficiencies increase by increasing investigated concentration of the investigated compounds. The causality factors in Table 4 are very close to theoretical values which according to EFM theory should guarantee the validity of Tafel slopes and corrosion current densities. Values of causality factors in Table 3 indicate that the measured data are of good quality. The standard values for CF-2 and CF-3 are 2.0 and 3.0, respectively. The deviation of causality factors from their ideal values might due to that the perturbation amplitude was too small or that the resolution of the frequency spectrum is not high enough also another possible explanation that the inhibitor is not performing very well. The obtained results showed good agreement of corrosion kinetic parameters obtained with the EFM, Tafel extrapolation and EIS methods.

Table 4 : Electrochemical kinetic parameters obtained by EFM technique for mild steel in the absence and presence of various concentrations of tweens in 0.5 M HCl at 25˚C

Comp.	Conc., M	i_{corr}, $\mu A\ cm^{-2}$	β_c, $mVdec^{-1}$	β_a, $mVdec^{-1}$	CF-2	CF-3	θ	IE %	CR, mmy^{-1}
Blank	00	454.8	59	67	1.42	2.16	-----	-----	176.4
Tween 20	20	262.0	89	102	1.91	2.98	0.424	42.4	101.6
	40	242.6	85	102	1.92	3.86	0.467	46.7	94.21
	60	234.3	85	103	1.92	3.09	0.485	48.5	90.88
	80	196.6	86	105	1.93	3.20	0.568	56.8	76.24
	100	180.2	86	105	1.94	2.86	0.604	60.4	69.90
Tween 60	20	151.7	92	97	2.41	2.90	0.666	66.6	58.84
	40	125.6	95	98	2.42	3.41	0.724	72.4	48.69
	60	115.2	94	102	1.79	2.75	0.748	74.8	44.67
	80	107.5	91	112	1.85	3.64	0.764	76.4	41.69
	100	104.9	92	103	1.82	2.90	0.769	76.9	40.68



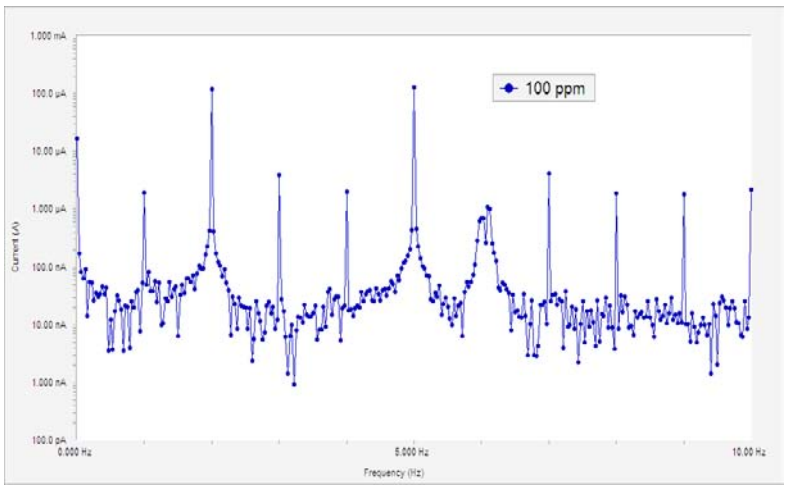


Figure 5 : EFM spectra for mild steel in the absence and presence of different concentrations of tween 20 in 0.5 M HCl

e) Adsorption isotherms

Organic molecules inhibit the corrosion process by the adsorption on metal surface. Theoretically, the adsorption process can be regarded as a single substitutional process in which an inhibitor molecule, I, in the aqueous phase substitutes an "x" adsorbed on the metal surface [44,45] vis,

$$I_{(aq)} + xH_2O_{(sur)} \longrightarrow I_{(sur)} + xH_2O_{(aq)} \quad (5)$$

Where x is known as the size ratio and simply equals the number of adsorbed water molecules replaced by a single inhibitor molecule. The adsorption depends on the structure of the inhibitor, the type of the metal and the nature of its surface, the nature of the corrosion medium and its pH value, the temperature and the electrochemical potential of the metal-solution interface. Also, the adsorption provides information about the interaction among the adsorbed molecules themselves as well as their interaction with the metal surface.

The values of surface coverage, θ, for different concentration of the studied compound at different temperatures have been used to explain the best isotherm to determine the adsorption process.

By far the results of investigated Tweens were best fitted by Langmuir adsorption isotherm. Figures 6 and 7 show the plotting of C/θ against C at different temperatures for Tween 20 and 60, respectively. These plots gave straight lines with unit slope indicating that the adsorption of investigated Tweens on mild steel surface follows Langmuir adsorption isotherm [46]:

$$C / \theta = 1/ K + C \quad (6)$$

Where C is the concentration of inhibitor, θ the fractional surface coverage and K is the adsorption

equilibrium constant related to the free energy of adsorption ΔG^{o}_{ads} as [47].

$$K = 1/ 55.5 \exp (-\Delta G^{o}_{ads}/ RT) \quad (7)$$

Where R is the universal gas constant, T is the absolute temperature. The value 55.5 is the concentration of water on the metal surface in mol/ L.

The calculated ΔG^{o}_{ads} values, using Eq. (7), were also given in Table 5. ΔG^{o}_{ads} is expressed in kJ mol^{-1} of Org_{ads}. The negative values of ΔG^{o}_{ads} ensure the spontaneity of the adsorption process and the stability of the adsorbed layer on the mild steel surface. It is well known that values of ΔG^{o}_{ads} of the order of 40 kJ mol^{-1} or higher involve charge sharing or transfer from the inhibitor molecules to metal surface to form coordinate type of bond (chemisorption); lower indicate a physisorption [48,49].

The calculated ΔG^{o}_{ads} values are in the range 38.6-41.9 kJ mol^{-1} indicates, therefore, that the adsorption mechanism of the investigated compounds on mild steel in 0.5 M HCl solution is a simple physical adsorption. The higher negative values of ΔG°_{ads} indicate that these inhibitors are strongly adsorbed on the mild steel surface. Moreover, $|\Delta G^{\circ}_{ads}|$ of investigated compounds decreases in the order Tween 60 > Tween 20.This is in good agreement with the ranking of inhibitors efficiency obtained from the different investigated techniques. The higher values of K for tween 20 and 60 are $1.04x10^{5}$, $1.71x10^{5}$ M^{-1} respectively; indicate stronger adsorption on the mild steel surface in 0.5 M HCl solution. The strong interaction of inhibitor with mild steel surface can be attributed to the presence of O atoms and л-electrons in the inhibitor molecules. Lagrenee et al [50] have reported that the higher K value (> 100 M^{-1}), the stronger and more stable adsorbed layer is forming which results in the higher inhibition efficiency.

Figure 6. Langmuir adsorption isotherms for tween 20 for corrosion of mild steel in 0.5 M HCl at different temperatures

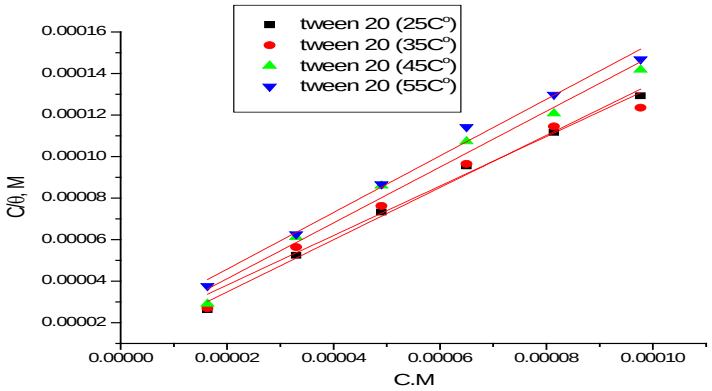


Figure 6 : Langmuir adsorption isotherms for tween 20 for corrosion of mild steel in 0.5 M HCl at different temperatures

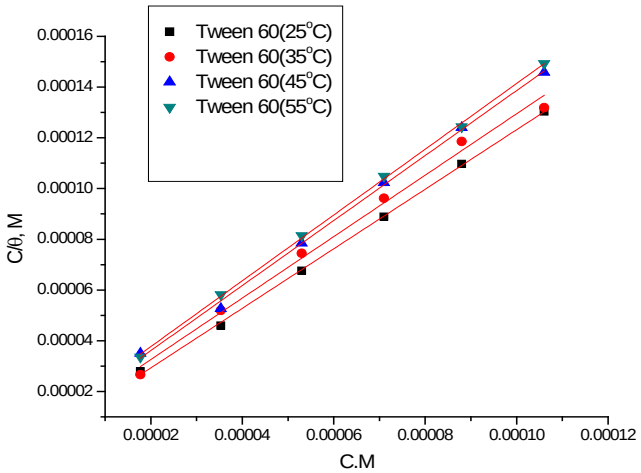


Figure 7 : Langmuir adsorption isotherms for tween 60 for corrosion of mild steel in 0.5 M HCl at different temperatures

f) Thermodynamic parameters

Thermodynamic parameters are important in studying the inhibitive mechanism. The values of enthalpy of adsorption, ΔH^{o}_{ads} and entropy of adsorption, ΔS^{o}_{ads} were obtained from the plot of ΔG^{o}_{ads} versus T and from the basic thermodynamic equation (8):

$$\Delta G^{o}ads = \Delta H^{o}ads - T\,\Delta S^{o}ads \quad (8)$$

ΔH^{o}_{ads} obtained were -10.9 and -18.9 kJ mol^{-1} for Tween 20 and 60 respectively. The negative sign of ΔH^{o}_{ads} obtained indicates the exothermic nature of the corrosion process [51] which indicates that % IE decreases with increasing the temperature. Generally, an exothermic process signifies either physisorption or chemisorption while endothermic process is attributable unequivocally to chemisorption [52]. In an exothermic process, physisorption is distinguished from chemisorption by considering the absolute value of a physisorption process is lower than 42 kJ mol^{-1} while the adsorption heat of a chemisorption process approaches 100 kJ mol^{-1} [53]. In the present case, the absolute value of the heat of adsorption is lower than 42 kJ mol^{-1} approaching the typical value of physisorption. The values obtained for ΔS^{o}_{ads} were 90 and 70 J $mol^{-1}K^{-1}$ for Tween 20 and 60 respectively. The negative values of ΔS^{o}_{ads} mean that the process of adsorption is accompanied by decrease in entropy. It might be explained as follows: before the adsorption of Tweens onto the mild steel surface, the chaotic degree of mild steel surface was high, but when inhibitor molecules were orderly adsorbed onto the mild steel surface, as a result, a decrease in entropy [54].

g) Effect of temperature

The effect of temperature on the rate of corrosion of mild steel in 0.5 M HCl containing different concentration from tween 20 or tween 60 was tested by potentiodynamic polarization measurements over a temperature range from 25 to 55°C.

The effect of increasing temperature on the corrosion rate (i_{corr}) and IE obtained from potentiodynamic polarization measurements.

The results revealed that, on the increasing solution temperature there is an increase of i_{corr} while IE decrease for all compound used. The activation energy (E^{*}_{a}) of the corrosion process was calculated using Arrhenius equation:

$$k = A \exp(-Ea^{*}/RT) \quad (9)$$

Where k is the rate of corrosion, A is the Arrhenius constant, R is the gas constant and T is the absolute temperature.

Figure 8 present the Arrhenius plot in the presence of 40 ppm from investigated Tweens. E^{*}_{a} values determined from the slopes of these linear plots are 21.8, 31.3 and 35.1 kJ $mol^{-1}$for blank, tween 20 and tween 60 respectively. The linear regression (R^2) is close to 1 which indicates that the corrosion of mild steel in 0.5 M HCl solution can be elucidated using the kinetic model. The values of E^{*}_{a} for inhibited solution is higher than that for uninhibited solution, suggesting that dissolution of mild steel is slow in the presence of tween and can be interpreted as due to physical adsorption [55]. It is known from Eq. 6 that the higher E^{*}_{a} values lead to the lower corrosion rate. This is due to the formation of a film on the mild steel surface serving as an energy barrier for the mild steel corrosion [56].

Enthalpy and entropy of activation (ΔH^{*}, ΔS^{*}) of the corrosion process were calculated from the transition state theory:

$$(i_{corr}) = (RT/Nh) \exp(\Delta S^{*}/R) \exp(-\Delta H^{*}/RT) \quad (10)$$

Where h is Planck's constant and N is Avogadro's number. A plot of log (i_{corr}/ T) vs. 1/ T for mild steel in 0.5 M HCl at 80 ppm from investigated compounds, gives straight lines as shown in Figure 9. Values of ΔH^{*} are, 8.5, 12, 14.2 kJ $mol^{-1}$for blank, tween 20 and tween 60, respectively and these values are positive. This indicates that the corrosion process is an endothermic one. The entropy of activation is large and negative and in the range 97.3 to 110.9 $Jmol^{-1}K^{-1}$. This implies that the activated complex represents association rather than dissociation step , indicating that a decrease in disorder takes place, going from reactants to the activated complex [52].

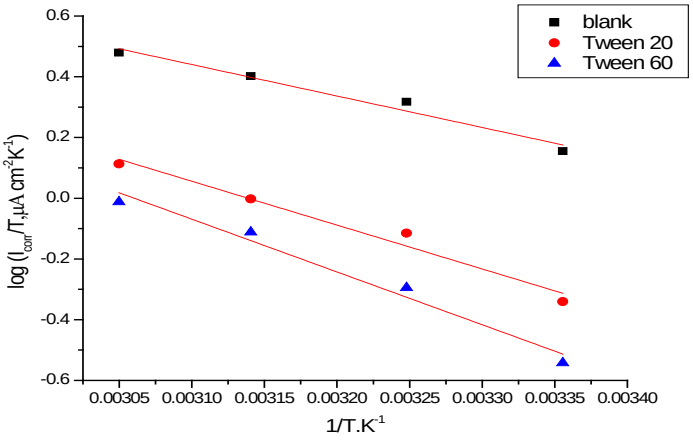


Figure 8 : Log i (corrosion rate) – 1/T curves for mild steel dissolution in 0.5 M HCl in the absence and presence of 80 ppm of the investigated compounds

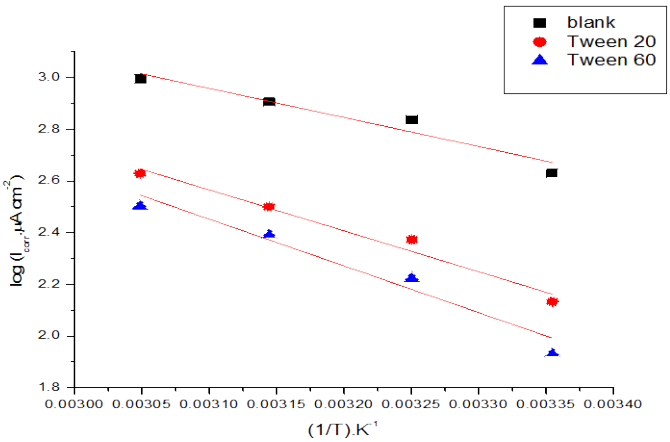


Figure 9 : Log i/T (corrosion rate)/T – 1/T curves for mild steel dissolution in 0.5 M HCl in the absence and presence of 80 ppm of the investigated compounds

h) Scanning electron microscopy (SEM) studies

Scanning electron microscopy (SEM) was employed to study the surface morphology of mild steel surface. The sample was studied after etching and \ or 24 hrs immersion in the test solution, in both cases after mechanical polishing. Figure [10(a)] reveals the microstructure of polished mild steel before placing it in the test solution. The scan shows that a solid and homogeneous surface is found. Figure [10 (b)] illustrates the effect of 0.5 M HCl on mild steel sample after 24 hrs immersion at 25°C, it appears that the presence of general corrosion (a large number of vacuoles with different sizes). Figure [10 (c)] exhibit the effect of 100 ppm tween 20 in 0.5 M HCl, it obvious that the presence of thin protection layer on mild steel surface and the surface becomes more smoother than that appears in case of HCl acid alone. On comparing Figure [10 (d)] which illustrates the effect of 100 ppm tween 60 in 0.5 M HCl at 25°C on mild steel sample, it appears that the disappearance of vacuoles and the formation of an adsorbed film on mild steel surface due to adsorption of the tweens lead to high corrosion inhibition at this concentration. This confirms with the previous results obtained from electrochemical studies.

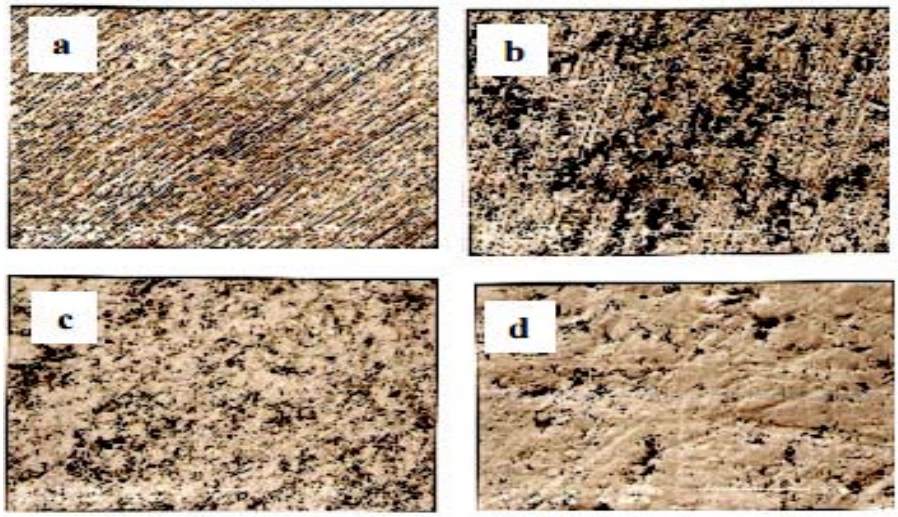


Figure 10 : SEM photographs of mild steel, (a) before immersion, (b) after corrosion in 0.5 M HCl, (c) in presence of 100 ppm tween 20 and (d) in presence of 100 ppm tween 60 in 0.5 M HCl solution at 25°C

i) Mechanism of corrosion inhibition

The adsorption may be the result of one or more of three types of interactions [53] namely; electrostatic attraction between charged molecules and charged metal, coordination of the unshared pairs of electron on the molecule to the metal atom, and involvement ofπ electrons of the inhibitor molecule in coordination process. From the observations drawn from the different methods, one can conclude that the inhibitor is adsorbed on mild steel surface forming a barrier film and protect substrate against corrosion in 0.5 M HCl solution. The inhibitive action of Tween compounds could be attributed to the adsorption of their molecules on mild steel surface forming a barrier between the bar metal and the corrosive environment. The surface activity of Tween compounds as well as the presence of function groups, such as carbonyl group, in their structures facilitates such adsorption. The surfactant molecules adsorb on the metal surface via their function groups, by van der Waals force. In addition, the main hydrophilic part, $CHO(CH_2CH_2O)_nCH_2CH_2$-OH, of Tweens 20 and 60 attacks the mild steel surface while the main hydrophobic part $CHCH_2OCO(CH_2)_{10}CH_3$ of Tween 20 and $CHCH_2OCO(CH_2)_{16}CH_3$ for Tween 60 extend to the solution face and they repel the aqueous aggressive anion away from the metal surface and therefore inhibit the corrosion reaction. When Tweens adsorbed on metal surface, coordinate bond might be formed by partial transference of electrons from the polar atom (O atom) of Tweens to the metal surface.

In general, inhibition of different tween compounds depends on their structures. The inhibition efficiency increases in the following order: T 20 < T 60.

This sequence reflects the effect of type of the fatty acid included in the tween formula, on their inhibitive action, since all tween compounds are mainly polyoxyethylene sorbitane combined with alkyl chain of different fatty acids which determine the tween number. Now, one can rewrite the above sequence, according the alkyl chain as following. Laurate (C12) < stearate (C18)

This new sequence illustrates the effects of both hydrocarbon length and presence of a double bond in the inhibitor structure. Thus, the inhibition efficiency increases as the number of carbon atoms in the alkyl chain increases.

IV. Conclusions

It was found that the percentage inhibition efficiency depends on the concentration, temperature and chemical structure of Tweens and the inhibition efficiency is in the order: Tween 60 > Tween 20. The inhibition efficiency increases as the length of the tween hydrocarbon chain is increased. Polarization curves demonstrate that the examined Tweens behave as mixed type inhibitors. The results of EIS indicate that the double layer capacitance decreases with respect to the blank solution when these inhibitors are added; this fact may be explained on the basis of adsorption of these inhibitors on mild steel surface. The adsorption of investigated Tweens on mild steel surface in HCl solution follows Langmuir adsorption isotherm. The negative values of ΔG^{o}_{ads} show the spontaneity of the adsorption of Tweens on mild steel surface. The percentage inhibition efficiency of Tweens obtained from the weight loss, potentiodynamic polarization curves, EIS and EFM techniques are in good agreement.

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Development and Properties of Hybrid Composites

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Abstract- The Flexural and tensile properties and Scanning electron Microscope analysis of Aramide/Onion/Glass fibers Reinforced Epoxy Hybrid composites were studied. The effect of alkali treatment of the Aramide/Onion/Glass fibers on these properties was also studied. These properties found to be higher when alkali treated glass fibers were used in the hybrid composites. The elimination of amorphous hemi-cellulose with alkali treated leading to higher crystallinity of the Aramide/ Onion/Glass fibers with alkali treatment may be responsible for these observations. The author investigated the interfacial bonding between Aramide/Onion/Glass reinforced epoxy composites. The effect of alkali treatment on the bonding between Aramide/Onion/Glass composites was also studied.

Keywords: aramide fiber, glass fiber, onion fiber, epoxy resin, SEM flexural properties, chemical resistance, tensile properties, hybrid composites.

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Development and Properties of Hybrid Composites

G. Meenambika Bai [α] & H. Raghavendra Rao [σ]

Abstract- The Flexural and tensile properties and Scanning electron Microscope analysis of Aramide/Onion/Glass fibers Reinforced Epoxy Hybrid composites were studied. The effect of alkali treatment of the Aramide/Onion/Glass fibers on these properties was also studied. These properties found to be higher when alkali treated glass fibers were used in the hybrid composites. The elimination of amorphous hemi-cellulose with alkali treated leading to higher crystallinity of the Aramide/ Onion/Glass fibers with alkali treatment may be responsible for these observations. The author investigated the interfacial bonding between Aramide/Onion/Glass reinforced epoxy composites. The effect of alkali treatment on the bonding between Aramide/Onion/Glass composites was also studied.

Keywords: aramide fiber, glass fiber, onion fiber, epoxy resin, SEM flexural properties, chemical resistance, tensile properties, hybrid composites.

I. Introduction

Several studies on the composites made from epoxy matrix and natural fibers onion, jute, wood, banana, sisal, cotton, coir and wheat straw were reported in the literature. Jindal (1) reported the development of bamboo fiber reinforced plastic composites using araldite (CIBA CY 230) resin as matrix. Though bamboo is extensively used as a valuable material from times immemorial (because of its high strength and low weight), the studies on this fiber reinforced plastics re meager. In the present work , the aramide, onion & glass fiber reinforced high performance epoxy hybrid composites were developed and their Flexural and tensile properties with fiber content were studied. The author investigated the interfacial bonding between Aramide, onion, glass reinforced epoxy composites. The effect of alkali treatment on the bonding between Aramide, onion, glass composites was also studied.

II. Materials and Methods

a) Materials

High performance epoxy resin LY 556 and the curing agent hardener HY 951 system were used as the matrix. Onion fiber were procured from local area. Some of these fibers were soaked in 1% NaOH solution for 30 min. to remove any greasy materials and hemi-cellulose, washed thoroughly in distilled water and dried under the sun for one week. The fibers with a thickness of 0.3mm were selected in the mat form. The glass chopped strand mat was used in making the hybrid composite percentage.

b) Preparation of mould

For making the composites, a moulding box was prepared with glass with 200mmx200mmx3mm mould (length x width x thickness)

III. Preparation of the Composite and The test Specimens

The mould cavity was coated with a thin layer of aqueous solution of poly vinyl alchol (PVA) which acts as a good releasing agent. Futher a thin coating of hard wax was laid over it and finally another thin layer of PVA was coated. Each coat was allowed to dry for 20 min at room temperature. A 3mm thick plate was made from the epoxy and hardener taken in the ratio of 100 and 10 parts by weight respectively. Then the moulding box was loaded with the matrix mixture and onion & glass fiber in random orientation (with varying percentage) and was placed in a vacuum oven which was maintained at 100oc for 3 hours to complete curing. After curing the plate was removed from the moulding box with simple tapering and it was cut in to samples for flexural test with dimensions of 150mmx20mmx3mm are cut as per ASTM specifications. For comparison sake the specimen for matrix material were also prepared in similar lines. For Scanning electron microscope analysis the cryogenically cooled and fractured specimen surfaces were gold coated and the fractures surface was observed using scanning electron microscope.

IV. Tensile and Flexural Load Measurement

The tensile and flexural modulus were determined using M/S Instron 3369 Model UTM. The cross head speed for flexural test was maintained at 10mm/min respectively. In each case 5 samples were tested and the average values are reported.

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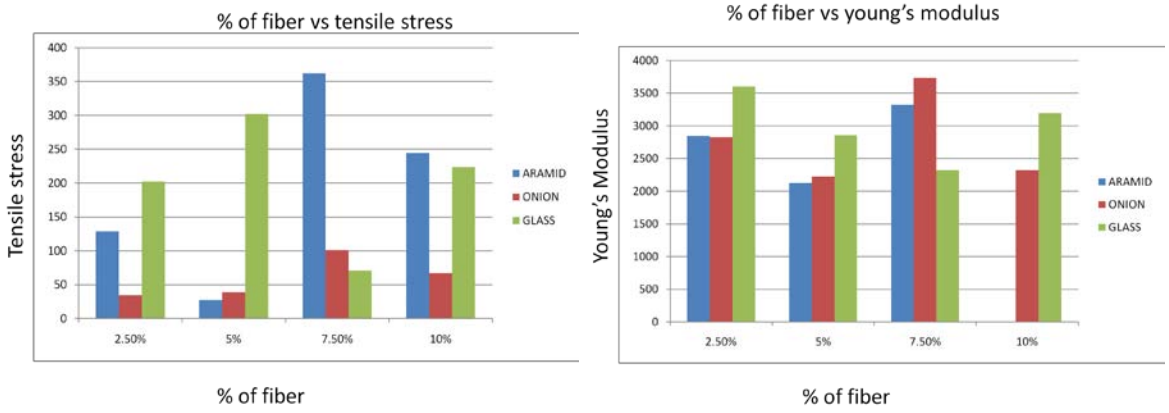
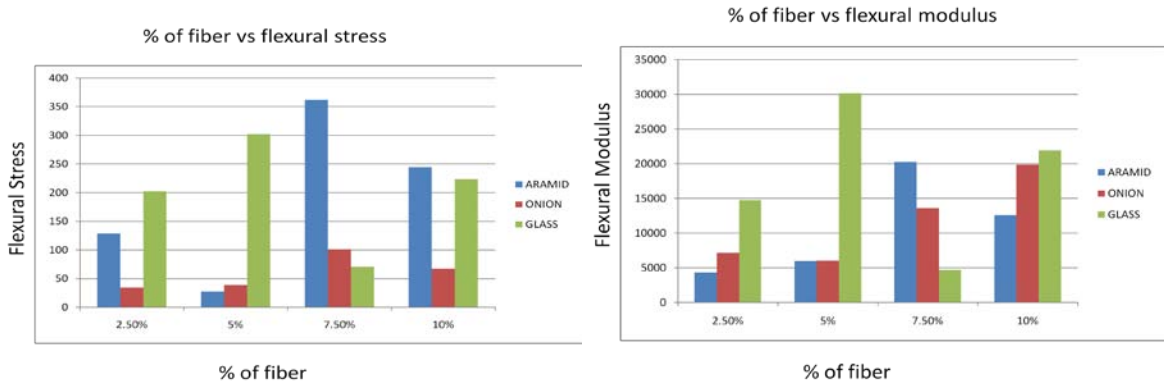


The variation of Stress with the ratio of Aramide, onion, Glass fiber Reinforced Epoxy composites



The variation of Flexural modulus with ratio of Aramide, Glass, onion fibers reinforced Epoxy composites

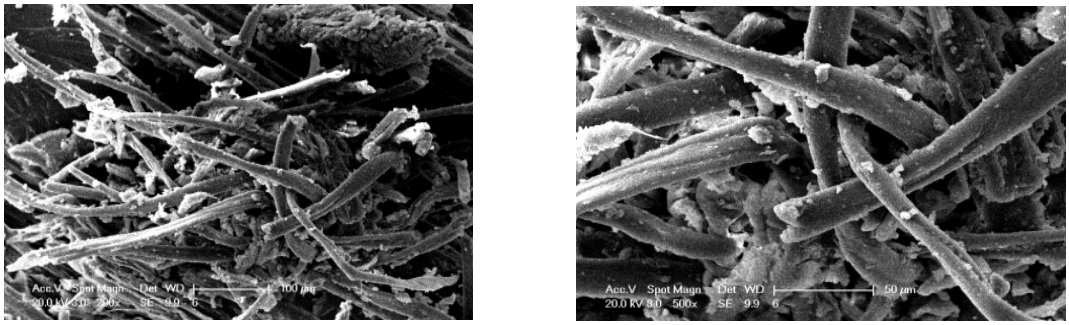
V. Epoxy Composites

a) Sem Analysis

To probe the bonding between the reinforcement and matrix, the Scanning electron micrograms of fractured surfaces of aramide, onion, glass reinforced epoxy composites were recorded. These micrograms were recorded at different magnifications and regions. The analysis of the micrograms of the composites prepared under different conditions is presented in the following paragraphs.

b) Untreated Onion Fiber

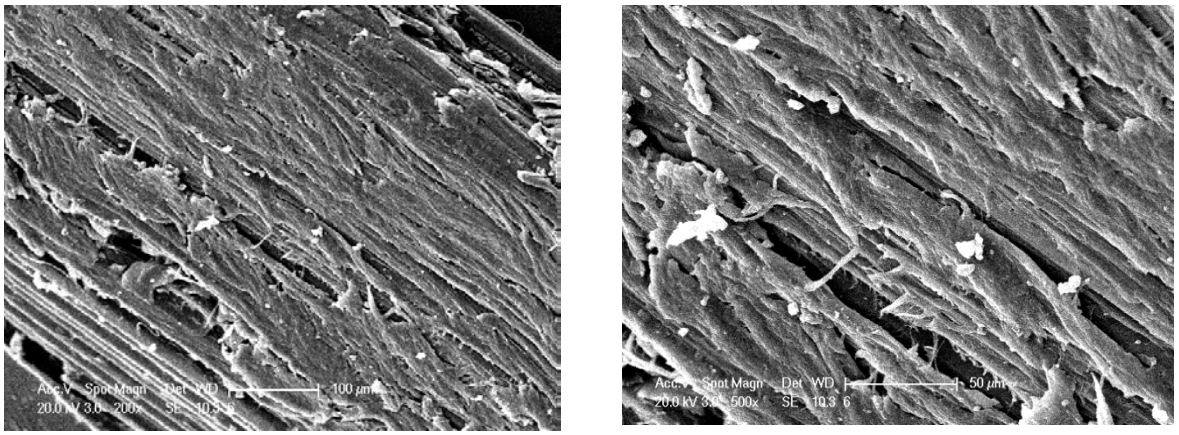
The micrograms of fractured surfaces of untreated onion fiber are presented in figure 2 (a), (b), (c&d). Figure 2 (a) & (b) represents the fractograms at two regions with a magnification of 100X. Figure 2(c) & (d) and the fractograms at these regions at magnification of 200X. From all these micrograms it is evident that fiber pullout is observed, indicating a poor bonding between the fibers. When the interfacial bonding is poor, the mechanical properties of the composites will be inferior. All the mechanical properties of the glass/onion fiber composites studied indicate that these properties are the least for these composites with untreated onion fibers. The poor adhesion is indicated in figure 2 supports this observation.



(a) (b)

Figure : SEM of untreated Onion fiber (a) and (b) at two regions 100x magnification

The fractograms of alkali treated bamboo fiber are presented in fig 3(a), (b).these fractograms were recorded at two different regions and 100X and 200X magnifications. From these micrograms it is clearly evident that the surface of the fiber becomes rough on alkali treatment. The elimination of hemi-cellulose from the surface of the bamboo fiber may be responsible for the roughening of the surface. Here, though the bonding is improved, fiber pullout is reduced. Thus the alkali treatment improved the bonding. This is in accordance with the mechanical properties of these composites.



(a) (b)

Figure : SEM of treated Onion fiber (a) and (b) at two regions 100x magnification

VI. Chemical Resistance of Composites

The chemical resistance of the composites was studied as per ASTM D 543-87 method. For chemical resistance test, the acids namely concentrated hydrochloric acid (10%), concentrated nitric acid (40%) and glacial acetic acid (8%), the alkalis namely aqueous solutions of sodium hydroxide (10%), ammonium hydroxide (10%) and sodium carbonate (20%) and the solvents- Benzene, carbon tetra chloride, toluene and water were selected. In each case, ten pre-weighted samples were dipped in the respective chemicals under study for 24 hours, removed and immediately washed thoroughly with distilled water and dried by pressing them on both sides by filter papers. The final weight of the samples and % weight loss/gain was determined. The resistance test was repeated for ten samples in each case and the average values reported.

Chemicals	Matrix	Composite
40 % nitric acid	+0.2876	+0.27541
10% Hydrochloric acid	+0.9365	+0.35491
8% Acetic acid	+0.3365	+2.4679
10% sodium hydroxide	-0.4761	-2.2191
20% sodium carbonate	+0.747	-3.7756
10% Ammonium Hydroxide	- 0.3243	-2.9985
Benzene	-1.321	-1.346
Toluene	-0.491	-2.340

Carbon tetrachloride	-1.124	+4.4858
Water	-1.112	-1.634

VII. Conclusion

The hybrid composites of onion/glass fiber reinforced epoxy were made and their Flexural properties and SEM analysis studied. The effect of alkali treatment of the bamboo fibers on these properties was studied. These hybrid composites were found to exhibit good Flexural properties. The hybrid composites with alkali treated onion fibers were found to possess higher flexural properties. The elimination of amorphous weak hemi-cellulose components from the Bombay fibers on alkali treatment may be responsible for this behavior.

VIII. Results and Discussion

The variation of Flexural strength with the ratio of percentage glass/onion fiber in these composites is presented in fig-1, fig-2. In this case also the hybrid composites are found to have good Flexural properties. In the case of maximum strength, the values vary between 60 to 213 MPa. The Flexural strength of these composites are found to be enhanced when alkali treated bamboo fibers were used in the hybrid composites. Similarities observation was made by Varada Rajulu et al (2-9) and Srinivasulu et al (10) in the case of some onion composites and polymer coated onion fibers.

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Key points to remember:

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A purpose of organizing a research paper is to let people to interpret your effort selectively. The journal requires the following sections, submitted in the order listed, each section to start on a new page.

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What to keep away from

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The page length of this segment is set by the sum and types of data to be reported. Carry on to be to the point, by means of statistics and tables, if suitable, to present consequences most efficiently.You must obviously differentiate material that would usually be incorporated in a study editorial from any unprocessed data or additional appendix matter that would not be available. In fact, such matter should not be submitted at all except requested by the instructor.

Content

- Sum up your conclusion in text and demonstrate them, if suitable, with figures and tables.
- In manuscript, explain each of your consequences, point the reader to remarks that are most appropriate.
- Present a background, such as by describing the question that was addressed by creation an exacting study.
- Explain results of control experiments and comprise remarks that are not accessible in a prescribed figure or table, if appropriate.
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What to stay away from

- Do not discuss or infer your outcome, report surroundings information, or try to explain anything.
- Not at all, take in raw data or intermediate calculations in a research manuscript.
- Do not present the similar data more than once.
- Manuscript should complement any figures or tables, not duplicate the identical information.
- Never confuse figures with tables - there is a difference.

Approach

- As forever, use past tense when you submit to your results, and put the whole thing in a reasonable order.
- Put figures and tables, appropriately numbered, in order at the end of the report
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Figures and tables

- If you put figures and tables at the end of the details, make certain that they are visibly distinguished from any attach appendix materials, such as raw facts
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- In spite of position, each table must be titled, numbered one after the other and complete with heading
- All figure and table must be adequately complete that it could situate on its own, divide from text

Discussion:

The Discussion is expected the trickiest segment to write and describe. A lot of papers submitted for journal are discarded based on problems with the Discussion. There is no head of state for how long a argument should be. Position your understanding of the outcome visibly to lead the reviewer through your conclusions, and then finish the paper with a summing up of the implication of the study. The purpose here is to offer an understanding of your results and hold up for all of your conclusions, using facts from your research and generally accepted information, if suitable. The implication of result should be visibly described. Infer your data in the conversation in suitable depth. This means that when you clarify an observable fact you must explain mechanisms that may account for the observation. If your results vary from your prospect, make clear why that may have happened. If your results agree, then explain the theory that the proof supported. It is never suitable to just state that the data approved with prospect, and let it drop at that.

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- You may propose future guidelines, such as how the experiment might be personalized to accomplish a new idea.
- Give details all of your remarks as much as possible, focus on mechanisms.
- Make a decision if the tentative design sufficiently addressed the theory, and whether or not it was correctly restricted.
- Try to present substitute explanations if sensible alternatives be present.
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- Recommendations for detailed papers will offer supplementary suggestions.

Approach:

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Methods and Procedures	Clear and to the point with well arranged paragraph, precision and accuracy of facts and figures, well organized subheads	Difficult to comprehend with embarrassed text, too much explanation but completed	Incorrect and unorganized structure with hazy meaning
Result	Well organized, Clear and specific, Correct units with precision, correct data, well structuring of paragraph, no grammar and spelling mistake	Complete and embarrassed text, difficult to comprehend	Irregular format with wrong facts and figures
Discussion	Well organized, meaningful specification, sound conclusion, logical and concise explanation, highly structured paragraph reference cited	Wordy, unclear conclusion, spurious	Conclusion is not cited, unorganized, difficult to comprehend
References	Complete and correct format, well organized	Beside the point, Incomplete	Wrong format and structuring

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