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Highlights

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Discovering Thoughts, Inventing Future

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Models of Turbulent Transport and Transfer of Disperse Phase Mass in Liquids

By Laptev A. G. & Farakhov T. M.

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Abstract- Using the theory of turbulent migration of particles and boundary layer models, expressions for calculation of particle transport coefficients and of mass transfer within the continuous phase are obtained. Various cases of turbulent direct flow of particles through the carrier turbulent medium are considered: smooth and rough channel; channel filled with fine random packing and apparatuses with mechanical mixing. Examples of calculation of mass transfer from droplets and solid particles are included and their agreement with experimental data of other authors is demonstrated.

Keywords: turbulence, direct flow, momentum transfer, mass transfer, mixing, extraction, dissolution.

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МОДЕЛИ ТУРБУЛЕНТНОГО ПЕРЕНОСА И МАССООТДАЧИ ДИСПЕРСНОЙ ФАЗЫ В ЖИДКОСТЯХ

Models of Turbulent Transport and Transfer of Disperse Phase Mass in Liquids

Лаптев А. Г.^а & Фарахов Т. М.^о

Laptev A. G. ^a & Farakhov T. M. ^o

Abstract- На основе применения теории турбулентной миграции частиц и моделей пограничного слоя получены выражения для расчета коэффициентов переноса частиц и массоотдачи в сплошной фазе. Рассмотрены различные случаи турбулентного прямотока частиц в несущей турбулентной среде: гладком и шероховатом канале; канале с мелкой неупорядоченной насадкой и аппаратах с механическим перемешиванием. Приведены результаты расчетов коэффициентов массоотдачи от капель и твердых частиц и показано согласование с экспериментальными данными различных авторов.

Abstract- Using the theory of turbulent migration of particles and boundary layer models, expressions for calculation of particle transport coefficients and of mass transfer within the continuous phase are obtained. Various cases of turbulent direct flow of particles through the carrier turbulent medium are considered: smooth and rough channel; channel filled with fine random packing and apparatuses with mechanical mixing. Examples of calculation of mass transfer from droplets and solid particles are included and their agreement with experimental data of other authors is demonstrated.

Ключевые слова: Турбулентность, прямоток, перенос импульса, массоотдача, перемешивание, экстракция, равстворение.

Keywords: turbulence, direct flow, momentum transfer, mass transfer, mixing, extraction, dissolution.

I. Введение

ля более эффективного проведения процесса массообмена, например, при растворении частиц или экстрагировании каких либо компонентов, дисперсную фазу подают в несущий турбулентный поток. Это может быть пустутелый канал

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Author: Инженерно-внедренческий центр «Инжехим», Казань, Россия. e-mail: info@ingehim.ru или канал заполненный хаотичной насадкой или аппарат с механической мешалкой. Аналогично работают и проточные смесители. Определение аппарат с механической мешалкой. Аналогично работают и проточные смесители. Определение эффективности таких процессов возможно только при известных характеристиках переноса дисперсной фазы в объеме сплошной.

При теоретическом анализе всех форм движения частиц в турбулентном потоке обычно принимаются следующие предположения[14].

А.Диаметр частиц $d_{\rm q}$ мал по сравнению с масштабом несущих их пульсационных вихрей с масштабом ℓ : $d_{\rm q} << \ell$.

При таком предположении каждая частица совершает движение, оставаясь в пределах несущего вихря.

Отмеченному условию удовлетворяют частицы любой дисперсности т.е. высокодисперсные ($d_{\rm q}$ <1 мкм); тонкодисперсные ($1 < d_{\rm q} < 20$ мкм) и грубодисперсные ($20 < d_{\rm q} < 200$ мкм).

Б. Частицы имеют форму близкой к сферической, а в случае сильного отклонения от сферы вводится коэффициент формы. Полидисперсность частиц рассматривается пофракционно.

В. Кроме этого частицы

 а) не стесняют движение друг друга в х оде взаимных перемещений;

б) не соударяются, не коагулируют друг с другом;

в) не оказывают ощутимого влияния на турбулентные характеристики среды.

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II. Коэффициент Переноса Импульса

Для определения скорости турбулентной миграции частиц (коэффициента переноса) первоначально рассмотрим процесс переноса импульса в однофазной турбулентной среде.

Для проекции потока импульса (касательного напряжения трения) на ось *оу* в любой точке пограничного слоя справедливо следующее выражение:

$$\tau(y) = -v\rho \frac{dU}{dy} - \rho v_{\delta}(y) \frac{dU}{dy}, \qquad (1)$$

где v, v $_{\rm T}(y)$ - коэффициенты кинематической молекулярной и турбулентной вязкости, м²/с; y — поперечная координата,м; ρ — плотность среды,

 $\hat{e}\tilde{a}/\hat{l}^{3}$; U – скорость, \hat{l}/\tilde{n} .

На поверхности стенок, т.е. при y = 0, этот же поток можно записать в виде [12]: $\tau_{\tilde{n}\tilde{o}} = \rho \gamma \Delta U$, где γ — коэффициент переноса импульса, м/с; ΔU движущая сила переноса, м/с.

Введем относительный поток импуляса $* = \tau(y)/\tau_{cr}$. Тогда проведя соответствующую замену в уравнении (1), разделяя переменные, и записывая интеграл по толщине пограничного слоя, получим [7,12]:

$$\rho \int_{0}^{\infty} \frac{dU}{\tau_{\rm co}} = \int_{0}^{\delta} \frac{\tau^* dy}{v + v_{\rm o}(y)},\tag{2}$$

где δ – толщина пристенного слоя, м.

Из данных выражений запишем коэффициент импульсоотдачи

$$\gamma = \left[\int_{0}^{\delta} \frac{\tau^* dy}{v + v_{\delta}(y)} \right]^{-1}.$$
 (3)

Выражение (3) является достаточно общим и позволяет рассчитать коэффициент импульсоотдачи на основе коэффициентов молекулярной и турбулентной вязкости $v_{\tau}(y)$, а также относительного касательного напряжения τ (относительного потока импульса) в пристенном слое.

В явном виде коэффициент переноса импульса можно получить путем интегрирования выражения (3) с различными функциями коэффициентов турбулентного обмена $v_{\tau}(y)$, используя полуэмпирические модели.

В модели Ландау и Левича, подтвержденной Дайслером и Ханратти предполагается, что изменение турбулентной вязкости в вязком подслое пропорционально $D_{\rm T} \sim y^4$. На основании этого используются функции $v_{\rm T}(y)$ в форме

$$\frac{v_{\rm T}}{v} = 0.01\chi^4 \left(y^+\right)^4, \ y^+ < 5,$$
$$\frac{v_{\rm T}}{v} = 0.2 \ y^+ - 0.959, \ 5 < y^+ \le 30, \qquad (4)$$

$$\frac{v_{\rm T}}{v} = 0.4 y^+ -1$$
 при $y^+ > 30$,

где $y^+ = u_* y/v$ - безразмерная координата; $\chi=0.4$ - константа; u_* - динамическая скорость, м/с.

После численного интегрирования (3) с функциями (4) получено [10]

$$\gamma = \frac{u_*}{5.309 + 2.5 \ln(R_{\delta})},$$
(5)

где $R_{\delta} = u_* \delta / v$ - безразмерная толщина пограничного слоя.

С функциями турбулентного обмена из двуслойной модели Прандтля

$$\frac{v_{\circ}}{v} = 0, \quad y^+ \le 11.6,$$
 (6)

$$\frac{v_{\delta}}{v} = \chi y^+, \quad 11.6 < y^+ \le R_{\delta},$$
 (7)

после интегрирования (3) получено выражение

$$\gamma = \frac{u_*}{R_1 + \frac{1}{\chi} \ln(R_\delta / R_1)},\tag{8}$$

где $R_1 = u_* \delta_1 / v$ – безразмерная толщина вязкого подслоя, в модели Прандтля $R_1 = 11.6$; $\chi = 0.4$ – константа турбулентности.

Выполним проверку выражений (5) и (8). Для этого на основе гидродинамической аналогии запишем связь между коэффициентами переноса импульса и теплоотдачи[7,10]

$$\alpha = \gamma \rho c_p / \Pr^n, \tag{9}$$

где по Чилтону-Кольборну n = 0,66. Однако, как известно, для пластины и трубы экспериментально установлено n = 0,6-0,57.

Из выражений (5), (8) и (9) для пластины запишем число Нуссельта, учитывая, что $u_* = U_{\infty} \sqrt{C_f/2}$, C_f =0.074 $\text{Re}_{nn}^{-0.2}$ – коэффициент трения; $\text{Re}_{nn} = U_{\infty}L/\nu$ – число Рейнольдса для пластины U_{∞} – средняя скорость при $y > \delta$, ì / \tilde{n} ; L – длина пластины, м. Получим

$$Nu_{\tilde{i}\tilde{e}} = \frac{Re_{\tilde{i}\tilde{e}} \sqrt{\tilde{N}_f / 2} Pr^{0.43}}{5.309 + 2.5 \ln R_{\delta}},$$
 (10)

$$\operatorname{Nu}_{\mathrm{i}\breve{e}} = \frac{\operatorname{Re}_{\mathrm{i}\breve{e}} \sqrt{\tilde{N}_f / 2} \operatorname{Pr}^{0.43}}{R_1 + \frac{1}{\chi} \ln(R_\delta / R_1)}.$$
 (11)

Значение R_{δ} – найдем используя функцию для логарифмического профиля скорости при $y = \delta$ [12] $R_{\delta} = \exp[\chi(U_{\infty}/u_* - 5,5)].$

Расчеты по выражениям (10) и (11) удовлетворительно согласуются с известными опытными данным для пластины (±2-6 %). Таким образом, проверена адекватность уравнений математической модели переноса импульса и тепла для плоского турбулентного пограничного слоя.

Т.к. выражения (5), (8) дают одинаковые результаты, в дальнейших расчетах переноса импульса удобнее использовать выражение (8), т.к. оно содержит безразмерную толщину вязкого подслоя, которую можно корректировать при наличии возмущений в пограничном слое.

Далее рассмотрим канал с хаотичной мелкой насадкой (элементами). Характеристики пограничного слоя на стенке канала с насадкой отличаются от характеристик плоского пограничного слоя без возмущений на пластине и поэтому значение $R_1 \neq 11.6$. Для таких случаев получено выражение[7].

$$\widetilde{R}_{1} = 11.6 \frac{U_{\infty}}{u_{*}} \sqrt{C_{f_{\tilde{1}\tilde{e}}}} / 2,$$
 (12)

где C_{finn} - коэффициент трения при движении потока со скоростью \overline{U}_{∞} вдоль плоской пластины; u_* – динамическая скорость в канале с насадкой, $1/\tilde{n}$ [13].

Для каналов известна связь $C_{finn} = \lambda/4$, где λ -коэффициент трения в гладких пустотелых трубах; λ =0.316/Re₃^{0.25}; Re₃= $U_{cp}d_{3}/\nu$ (для эквивалентного канала $d=d_{3}$); $d_{\acute{y}}$ – эквивалентный диаметр насадки, м; $d_{\acute{y}} = 4\varepsilon_{\acute{n}\acute{a}}/\dot{a}_{v}$; $\varepsilon_{\acute{n}\acute{a}}$ – удельный свободный объем, λ^{3}/λ^{3} ; \dot{a}_{V} – удельная поверхность насадки, λ^{2}/λ^{3} . Из выражения (12) следует

$$\widetilde{R}_{1} = 11.6 \frac{u_{*\widetilde{I}\widetilde{e}}}{u_{*}} = 11.6 \sqrt{\frac{\tau_{\widetilde{n}\widetilde{o}\widetilde{i}\widetilde{e}}}{\tau_{\widetilde{n}\widetilde{o}}}},$$
(13)

где $u_{*_{\Pi\Pi}}$ и $\tau_{c_{\Pi}\Pi_{\Pi}}$ - динамическая скорость и касательное напряжение на пластине (в пустотелой трубе) при скорости среды U_{∞} .

Таким образом, корректировка R_1 производится путем отношения потоков импульса в плоском и в возмущенном пограничном слое при одинаковой средней скорости движения среды.

Среднюю толщину гидродинамического пограничного слоя на элементах насадки при условии стабилизированного течения найдем как $\delta = \varepsilon_{\rm cB}/a_{\rm v}$. Отсюда $\delta=0.25d_3$ и значение R_{δ}

$$R_{\delta} = \frac{0.25d_{\acute{y}}u_{\ast}}{v}.$$
 (14)

Приведенные выше выражения для коэффициентов переноса импульса справедливы для турбулентных однофазных сред или гетерогенных сред, когда плотности фаз практически одинаковые (например, две смешиваемые жидкости).

III. Коэффициент Переноса Частиц

Гидродинамические характеристики звешенных частиц в турбулентной среде отличаются гораздо большей сложностью и интенсивностью, чем в ламинарной. Расчеты показывают, что только достаточно крупные частицы в жидкостях (более 3-5 мм, в зависимости от гидродинамических условий среды и плотностей взаимодействующих фаз) не участвуют в турбулентных пульсациях среды. Для более мелких частиц при моделировании гидродинамических процессов в многофазных системах турбулентное частиц необходимо пульсационное движение учитывать.

В соответствии с теорией турбулентной миграции можно классифицировать частицы по группам на основании индекса инерционности $\omega_{\rm E}\tau_{\rm p}$, где $\omega_{\rm E}$ - угловая частота турбулентных низкочастотных пульсаций энергоемких вихрей, с⁻¹, $\tau_{\rm p}$ - время релаксации, с. По экспериментальным данным при значении $\omega_{\rm E}\tau_{\rm p}$ < 0.01, степень увлечения частиц турбулентными пульсациями среды достигает 100% ($\mu_{\rm p}^2$ =1), $\mu_{\rm p}^2$ = (1- $\omega_{\rm E}\tau_{\rm p}$)⁻¹[14].

Поэтому для степени увлечения для частиц практически полностью увлекаемых турбулентными пульсациями среды можно записать условие:

$$\omega_E \tau_p = \frac{\pi |\Delta \rho| d_{\div}^2 f}{9\mu_{\mathfrak{X}}} < 0.01.$$
(15)

Отсюдо получено [1] выражение для оценки диаметра частиц с учетом, что по Таунсенду $\omega_{\rm E} \approx u_*/(0.1R)$ и $\tau_{\rm p} = |\Delta \rho| d_{\rm q}^2/(18\mu_{\rm w})$, $|\Delta \rho| \neq 0$:

$$d_{\pm} < 0.3 \sqrt{\frac{\mu_{x}}{\pi |\Delta \rho| f}} = 0.134 \sqrt{\frac{R\mu_{x}}{|\Delta \rho| u_{*}}}, \qquad (16)$$

где $\omega_{\rm E} = 2\pi f$ – угловая частота энергоемких пульсаций, с⁻¹; f – частота турбулентных пульсаций среды, с⁻¹; R - радиус канала, м (для насадки $R = d_{g}/2$, м);

 $|\Delta \rho| = |\rho_{\text{m}} - \rho_{\text{q}}|$ - разностей плотностей; ρ_{q} - плотность частицы, кг/м³; d_{q} - диаметр частицы, м; μ_{m} - динамическая вязкость, Па·с.

При больших значениях индекса инерционности $\omega_{\rm E}\tau_{\rm p} > 100$ степень увлечения приближается к нулю ($\mu_{\rm p}^2 \approx 0$). По аналогии с (15) и (16) получаем выражения для частиц, не увлекаемых турбулентными пульсациями

$$\omega_E \tau_p = \frac{\pi |\Delta \rho| d_{\div}^2 f}{9\mu_{\mathfrak{X}}} > 100, \tag{17}$$

Отсюда имеем

$$d_{\div} > 30 \sqrt{\frac{\mu_{\mathfrak{X}}}{\pi |\Delta\rho| f}} = 13.4 \sqrt{\frac{R\mu_{\mathfrak{X}}}{|\Delta\rho| u_{\ast}}}.$$
 (18)

Из уравнений (16) и (18) следует, что частица, взвешенная в турбулентном потоке, тем точнее следует за пульсациями среды, чем меньше ее радиус и разность плотностей фаз, чем больше вязкость среды и ниже частота ее пульсаций.

Коэффициент турбулентной диффузии частиц

(при условии
$$0.134 \sqrt{\frac{R\mu_{*}}{|\Delta\rho|u_{*}}} < d_{\pm} < 13.4 \sqrt{\frac{R\mu_{*}}{|\Delta\rho|u_{*}}}$$
)

можно определить по выражению[14]

$$D_{\pm} = \frac{v_{\delta}}{1 + \omega_{\lambda} \tau_{p}}.$$
 (19)

Коэффициент скорости переноса чафиц _ч (турбулентной миграции) найдем путем интегрирования выражения (3) и аналогии $\beta_{q} = \gamma / (1 + \omega_{E} \tau_{p})$.

Броуновская диффузия частиц не учитывается, т.к. ее влияние на перенос в турбулентном потоке очень незначительно.

Получим

$$\beta_{\div} = \frac{\mu_{\ast}}{\left(1 + \omega_E \tau_p \left[\widetilde{R}_1 + \frac{1}{\chi} \ln\left(\widetilde{R}_{\delta} / \widetilde{R}_1\right)\right]\right]}.$$
 (20)

С использованием известной зависимости для идеального вытеснения потока, которая соответствует энергетической модели, эффективность турбулентного переноса дисперсных частиц к стенке канала или к поверхности хаотичной насадки имеет вид.

$$\eta = 1 - \exp\left(-\frac{\beta_{\div}F}{V_{\&}}\right), \qquad (21)$$

где N — число единиц переноса; F — поверхность канала или насадочных элементов, ì²; V_x — объемный расход сплошной среды, ì³ / \tilde{n} .

Для пустотелого канала расчет по выражению (21) дает долю частиц, котор ые за счет тур булентной миграции приблизились (осели) на стенки. Если в канале находится мелкая хаотичная насадка, то выражение (21) характеризует эффективность смешения т.к. распределение дисперсной сред. фазы у практически элементов обеспечит поверхности однородную концентрацию по поперечному сечению канала, если $\eta \rightarrow 1$ [13]. Задавая значение $F = a_y SH$, можно выбрать длину зоны турбулентного смесителя, где a_v – удельная поверхность насадки, \hat{i}^2/\hat{i}^3 ; S – площадь поперечного сечения канала, \hat{i}^2 ; Н- длина зоны смешения с насадкой, м.

IV. Коэффициент Массоотдачи

Рассмотрим процесс массоотдачи в сплошной фазе при движении капель или твердых частиц в турбулентном прямотоке.

Экспериментальные исследования [3] методом голографической интерферометрии характеристик диффузионного пограничного слоя в случае массообмена мелкодисперсных твердых частиц в турбулентной жидкости, позволили установить автомодельность профиля концентрации, характерную для ламинарного пограничного слоя, и наличие логарифмического профиля концентрации, присущего турбулентным пограничным слоям. Пульсации турбулизированного внешнего течения, проникая в вязкий подслой частиц, вызывают усиление процессов массопереноса. Все вышеперечисленное позволяет рассматривать пограничный слой на частицах как «псевдоламинарный».

В работах [5-8] предложены модели массопереноса, позволяющие учитывать влияние внешней турбулентности на поведение псевдоламинарного слоя.

В системах жидкость-жидкость число Шмидта Sc >> 1 и тогда диффузионное сопротивление локализуется в подслое, который называют вязким. В вязком подслое сочетается молекулярный перенос с турбулентной затухающей диффузией. На границе диффузионного подслоя принимается $D_{\rm T} \approx D$, где $D, D_{\rm T}$ – коэффициенты молекулярной и турбулентной диффузии компонента, м²/с.

Учитывая, что толщина вязкого подслоя на порядок меньше диаметра частицы (капли) примем модель плоского пограничного слоя.

Сопротивление переносу массы компонента в вязком подслое на капле записывается в виде [6,7].

$$\frac{1}{\beta} = \int_{0}^{\delta_{1}} \frac{dy}{D + D_{\delta}},$$
(22)

где β – коэффициент массоотдачи от капли (к капле) в сплошной фазе, м/с; δ_1 - толщина вязкого подслоя на капле, м; у - поперечная координата в пограничном слое, м.

После интегрирования (22) с функцией турбулентного обмена Левича $D_{\rm T} = u_* \delta_1 (y/\delta_1)^2$, для капель с подвижной поверхностью раздела фаз ($\mu^* \leq 1$), коэффициент массоотдачи получит вид [6].

$$\beta = \frac{2u_*}{\pi\sqrt{R_1Sc}},\tag{23}$$

и для капель с $\mu^* > 1$, а также твердых частиц при $D_{T} = u_* \delta_1 (y/\delta_1)^3$ имеем [6].

$$\beta = \frac{6\sqrt{3}z^{2}\overline{u}_{*}}{\sqrt{3}\ln\frac{(z+1)^{2}}{z(z-1)+1}} 6\operatorname{arctg}\frac{(2-z)}{z\sqrt{3}} + \pi, \quad (24)$$

где $R_1 = u_* \delta_1 / v$ - безразмерная толщина вязкого подслоя; \overline{u}_* - динамическая скорость на межфазной поверхности капли, м/с; v – коэффициент кинематической вязкости сплошной фазы, M^2/c ; $z = (R_1Sc)^{-0.333}$; Sc = v/D – число Шмидта; $\mu^* = \mu_d / \mu_c$ – отношение коэффициентов динамической вязкости дисперсной и сплошной фаз.

Значение *R*₁ для формулы (23) получено в виде [6].

$$R_1 = \left[\Delta u \left(\overline{u}_* \operatorname{arctg} \sqrt{R_1}\right)^{-1}\right]^2, \qquad (25)$$

а для формулы (24)

$$R_{1} = \left[\frac{6\sqrt{3}\Delta u}{\overline{u}_{*}}\left(k_{1} + k_{2} + \pi\right)^{-1}\right]^{3/2},$$
 (26)

где
$$\kappa_1 = \sqrt{3} \ln \frac{(\hat{A}+1)^2}{\hat{A}(\hat{A}-1)+1}$$
; $\kappa_2 = 6 \ arctg$

 $\frac{(2-\hat{A})}{\hat{A}\sqrt{3}}$; $B = R_1^{-0.333}$; $\Delta u = u_0 - u_{\tilde{a}\delta}$; u_0 - средняя

скорость капли (частицы) относительно жидкости, м/с; $u_{\tilde{a}\tilde{o}}$ – скорость на межфазной поверхности, м/с (для частицы $u_{\tilde{a}\tilde{o}} = 0$)

Согласно правилу аддитивности среднее касательное напряжение на поверхности капли будет равно.

$$\overline{\tau} = \tau_{\hat{e}} + \tau_{\hat{o}}, \qquad (27)$$

где τ_{κ} - касательное напряжение при свободном движении капли, Па; $\tau_{\rm T}$ - касательное напряжение на капле, вызванное турбулентными пульсациями из внешнего потока, Па.

Из выражения (27) средняя динамическая скорость на капле.

$$\overline{u}_* = \sqrt{\left(\tau_k + \tau_{\delta}\right)/\rho_x} , \qquad (28)$$

где $\tau_{\hat{e}} = u_*^2 \rho_{a}$ и выражение для расчета имеет вид [9].

$$\tau_k = 64 \,\rho_{\ast} \,\frac{\Delta u \,\nu}{4\,\delta + 1.6\,d_k}\,,\tag{29}$$

где d_k – средний диаметр капли, м.

Значение $\tau_{\rm T}$ найдем, используя среднюю объемную скорость диссипации энергии, выраженную через перепад давления в канале [2,6,11].

$$\tau_{\delta} = 4 \rho_{\ast} \left(\frac{\overline{\varepsilon}\nu}{\rho_{\ast}}\right)^{1/2} = 4 \rho_{\ast} \left(\frac{\Delta P W_{\tilde{n}\delta}\nu}{\rho_{\ast} H}\right)^{1/2}, \quad (30)$$

где \mathcal{E} - средняя скорость диссипации энергии, Вт/м³; ΔP – перепад давления, Па; $W_{\rm cp}$ - средняя скорость жидкости в канале, м/с; H - длина канала, м.

Записывая ΔP по выражению Дарси-Вейсбаха из (30) получим.

$$\tau_{\delta} = 4 \rho_{\mathfrak{x}} \left(\frac{\lambda W_{\tilde{n}\delta}^{3} v}{2 d} \right)^{1/2}, \qquad (31)$$

где λ – коэффициент гидравлического сопротивления канала, $\lambda = f(Re_d)$; d – диаметр канала, м; $Re_d = W_{cp} d/v$ – число Рейнольдса. По формуле Блазиуса $\lambda = 0,316 Re_d^{-0.25}$ при 4000 < $Re_d < 10^5$.Приближенно в интервале 10^4 < $\operatorname{Re}_d < 10^6$, $\lambda = 0,184 \operatorname{Re}_d^{-0.2}$

Среднюю скорость движения капли относительно внешней среды в турбулентном несущем потоке запишем как результирующую.

$$u_0 = \sqrt{u_k^2 + u_\varepsilon^2} , \qquad (32)$$

где u_k – скорость гравитационного движения, м/с; u_ε - средняя скорость, вызванная турбулентными пульсациями среды, м/с.

Значение u_{ε} рассматривается как скорость обтекания частицы турбулентными пульсациями порядка диаметра частицы, выраженную через величину диссипации энергии в канале.

$$u_{\varepsilon} \approx \left(d_{k}\overline{\varepsilon}\right)^{1/3} = \left(\frac{d_{k}\lambda W_{\tilde{n}\tilde{\delta}}^{3}}{2d}\right)^{1/3}$$
 (33)

Расчеты коэффициента массоотдачи (23) в турбулентном прямотоке в гладкой трубе при $Re_d = 10^4 \div 10^5$ показывают, что по сравнению со свободным гравитационным режимом [6] происходит незначительное повышение коэффициента массоотдачи β на 5-10%. Если в трубе имеются элементы интенсификации (например, кольцевая накатка), то

коэффициент массоотдачи повышается на 20-25 %. Значительное повышение коэффициентов массоотдачи (в 2 раза) наблюдается, если в трубу поместить мелкую нерегулярную насадку, например «Инжехим» [4].

Среднее касательное напряжение на поверхности нерегулярных насадочных элементов находится по выражению (Re₃>50) [11].

$$\tau_{\circ} = 4 \rho_{*} \left(\nu / d_{y} \right)^{2} \operatorname{Re}_{y}^{1,5} \left(\xi / 2 \right)^{0,5}, \qquad (34)$$

где $Re_{9} = W_{cp}d_{9} / v$ – число Рейнольдса для насадки; $d_{9} = 4\varepsilon_{ce}/a_{v}$ – эквивалентный диаметр насадки, м; ε_{ce} – удельный свободный объем насадки, м³/м³; a_{v} – удельная поверхность, м²/м³; ζ – коэффициент гидравлического сопротивления насадочного слоя [4].

При расчете коэффициента массоотдачи от капель в турбулентном прямотоке в канале с хаотичной насадкой предполагается, что дисперсная фаза не смачивает поверхность насадки. Если же дисперсная фаза хорошо смачивает поверхность элементов насадки, то необходимо учитывать дополнительную массоотдачу от пленки жидкости в прямотоке, что повысит эффективность экстракции.

V. Массоотдача При Перемешивании

Далее рассмотрим применение выше представленных уравнений для расчета массотдачи от капель и твердых частиц в аппарате с мешалкой при турбулентном режиме.

Определение средней скорости относительного движения дисперсных частиц в перемешиваемой среде является сложной задачей, которая пока не имеет точного аналитического решения. В работе [15] предлагается среднюю скорость движения частиц представлять как результирующую скоростей (аналогично (32)):

$$u_0 = \sqrt{u_k^2 + u_{\varepsilon}^2 + u_{\hat{1}\hat{a}\hat{o}}^2}, \qquad (35)$$

где *и*_{обт} – скорость проскальзывания:

$$u_{\hat{i}\hat{a}\hat{o}} = \sqrt{\frac{\pi \left|\Delta\rho\right| u_i d_{\hat{e}}}{6\xi_{\hat{e}} \rho_{x} d_{\hat{i}}}},$$
(36)

где $u_{\rm M}$ – скорость вращения конца лопасти мешалки, м/с: $u_{\rm M} = \pi n \, d_{\rm M}$, n – частота вращения, с⁻¹; $d_{\rm M}$ – диаметр мешалки, м ξ – коэффициент сопротивления при движении капли, $\xi_{\rm k} = f(Re_{\rm k})$.

Среднюю динамическую скорость на межфазной поверхности капли найдем по выражению [6,7]

$$\overline{u}_* = 2 \left(\frac{\overline{\varepsilon} v_x}{\rho_x} \right)^{1/4}, \qquad (37)$$

где $\overline{\varepsilon}$ – средняя массовая скорость диссипации энергии:

$$\overline{\varepsilon} = \frac{N}{Q_x} = \frac{K_N \rho_x n^3 d_i^5}{Q_x}, \qquad (38)$$

где N – мощность потребляемая на перемешивание, Вт; K_N - критерий мощности, зависящий от конструктивных характеристик мешалки и центробежного числа Рейнольдса; Q_M - объем жидкости в мешалке, M^3 .

Расчет показывает, что значение параметра R_1 (25) для капель находится в пределах $R_1 \approx 6-8$.

Результаты расчета коэффициента массоотдачи в сплошной фазе по уравнению (24) и сравнение с опытными данными [2] показано на рисунке 1. Эксперименты проводились в проточном смесителе диаметром D = 38 мм при перемешивании 2-х лопастной мешалкой с соотношениями диаметра мешалки, ширины лопасти и диаметра аппарата, равными соответственно 1 : 2; 1 : 10.

Для твер ых частиц и капель с большой вязкостью, относительно сплошной среды, коэффициенты массоотдачи в сплошной фазе можно определить по уравнению (24) с параметром (26). Проведенные расчеты безразмерной толщины вязкого подслоя (26) показывают, что $R_1 \approx 15$ -25.



Рисунок 1: Зависимость коэффициентов массоотдачи β в сплошной фазе от комплекса nd_1 при экстракции различных систем: 1 – расчет по уравнению (23); 2 – экспериментальные данные, обобщенные в работе [2].

593·10⁻⁶, м.

анионообменной смолы в растворе соляной кислоты.

Диаметр частиц анионообменной смолы – 30, 8·10⁻⁶;

Многие авторы изучают массоотдачу в сплошной фазе при растворении твердых частиц.

На рисунке 2 показана корреляция расчетных и экспериментальных данных [15] при растворении



Рисунок 2 : Корреляционная зависимость расчетных и экспериментальных значений коэффициента массоотдачи β от диссипации энергии ε: 1, 2 – расчет по уравнению (24); 3, 4 – экспериментальные данные [14]. Диаметр частиц (м): 1, 3 – 30,8 · 10⁻⁶; 2, 4 – 593 · 10⁻⁶

На рисунке приведено сравнение 3 рассчитанных по уравнениям модели И экспериментальных данных различных авторов, обобщенных в работе [2], при растворении твердых взвешенных частиц в аппаратах с мешалками.



Рисунок 3 : Зависимость комплекса от диссипации энергии: 1 – расчет по уравнению (24); 2 – экспериментальные данные [2]

Уравнения (23) и (24) проведены для аппаратов с механическим перемешиванием: $\text{Re}_{\mu} > 10^3$, $10^ ^4 < d_{\kappa} < 10^{-3}$, 500 < Sc < 1700. Расхождение с экспериментальными данными $\pm 15\%$.

VI. Основные Результаты и Выводы

В статье рассмотрены процессы переноса частиц (< 10⁻³,м) в жидкостях при турбулентном прямотоке, а также массо отдачи в сплошной фазе. На основе исплользования моделей пограничного слоя получены выражения для коэффициентов переноса импульса, а с применением теории турбулентной миграции частиц коэффициенты переноса частиц (скорости турбулентной миграции). Полученные выражения могут использоваться для расчета турбулентных прямоточных смесителей.

Далее рассмотрена массоотдача в сплошной фазе от капель и твер ых частиц в пр мотоке. Приведены уравнения для расчета коэффициентов массоотдачи и выражения для вычисления их характеристик. Показано влияние на коэффициенты массоотдачи при прямотоке в канале кольцевых накаток и мелкой неупорядоченной насадки. Кроме этого выполнены расчеты массоотдачи в аппаратах с механическим перемешиванием. Показанно удовлетворенное согласование с экспериментальными данными различных исследователей.

Полученные уравнения для коэффициентов турбулентного переноса и массоотдачи дисперсных частиц могут использоваться для расчетов широкого класса аппаратов химической технологии и энергетики.

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Removal of Phosphates and Chromates in a Multi-ion System

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Abstract- Because industrialization is increasing the number of manufacturing facilities in traditionally rural agricultural areas globally, ground and surface waters with mixed fertilizer and industry contaminants must be considered. This work characterizes the effect of nitrates, phosphates, and chromates on the removal of phosphates and chromates from contaminated water via ion exchange with calcined hydrotalcite, an inexpensive, synthetic double layer hydroxide. Isotherms for single ion removal are compared with those for multi-ion solutions at contaminant levels typical for contaminated ground and surface waters. In addition, fixed effects statistical analysis of variance, PROC GLM (SAS Version 9) was used to analyze main and interactive effects for target anion removal and Least Significance Difference t-test and Tukey's studentized range honest significant difference at $\alpha = 0.05$ were used a multiple comparison procedures to determine significant mean difference in treatments.

It was found that in a binary phosphate-chromate system, phosphate removal was only significantly reduced by higher chromate levels and this effect was magnified at higher phosphate levels. Chromate removal, on the other hand, was reduced at all phosphate levels. For the ternary system, phosphate removal was reduced by chromates, but not nitrates. However, the chromate-nitrate interaction was significant, as was the ternary interaction, particularly at higher phosphate levels. Chromate removal in the ternary system was impacted by the main effects for chromate, phosphate, and nitrate and also by all of the interactive effects. Hence, a multi-step ion exchange process would be required to remove all anions of concern in a mixed contaminant system.

Keywords: ion exchange, phosphate, chromate, nitrate, hydrotalcite, double layer hydroxide.

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REMOVALOFPHOSPHATESANDCHROMATESINAMULTIIONSYSTEM

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Removal of Phosphates and Chromates in a Multi-ion System

Patricia A. Terry ^a, David Dolan ^c, Matthew J. Maccoux ^p & McKinley Meyer ^a

Abstract-Because industrialization is increasing the number of manufacturing facilities in traditionally rural agricultural areas globally, ground and surface waters with mixed fertilizer and industry contaminants must be considered. This work characterizes the effect of nitrates, phosphates, and chromates on the removal of phosphates and chromates from contaminated water via ion exchange with calcined hydrotalcite, an inexpensive, synthetic double layer hydroxide. Isotherms for single ion removal are compared with those for multi-ion solutions at contaminant levels typical for contaminated ground and surface waters. In addition, fixed effects statistical analysis of variance, PROC GLM (SAS Version 9) was used to analyze main and interactive effects for target anion removal and Least Significance Difference t-test and Tukey's studentized range honest significant difference at $\alpha = 0.05$ were used a multiple comparison procedures to determine significant mean difference in treatments.

It was found that in a binary phosphate-chromate system, phosphate removal was only significantly reduced by higher chromate levels and this effect was magnified at higher phosphate levels. Chromate removal, on the other hand, was reduced at all phosphate levels. For the ternary system, phosphate removal was reduced by chromates, but not nitrates. However, the chromate-nitrate interaction was significant, as was the ternary interaction, particularly at higher phosphate levels. Chromate removal in the ternary system was impacted by the main effects for chromate, phosphate, and nitrate and also by all of the interactive effects. Hence, a multistep ion exchange process would be required to remove all anions of concern in a mixed contaminant system.

Keywords: ion exchange, phosphate, chromate, nitrate, hydrotalcite, double layer hydroxide.

I. INTRODUCTION

s the rate of industrialization increases globally along with suburban growth bringing factories and industry to rural areas, the concern for water contaminated with a mixture of industrial and agricultural wastes increasing. Many people in these is

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rural areas rely on untreated well water for domestic use. In both the developed and developing worlds, phosphate and nitrate nutrient contamination from agricultural practices have rendered wells unsafe for consumption, primarily because the presence of nitrates above 10 mgL⁻¹ in drinking water can lead to methemoglobinemia, or blue baby syndrome, which affects both humans and farm animals. Nitrates and, particularly, phosphates are both responsible for increased rates of eutrophic waters, which leads to potentially toxic algae blooms and reduced oxygen levels. These greatly reduce the health of aqueous ecosystems, creating dead zones in many waterways globally. Phosphate levels as low as 0.025 mgL⁻¹ can accelerate eutrophication and, because it is the limiting for eutrophication. the United factor States Environmental Protection Agency, USEPA, recommends levels below 0.1 mgL⁻¹ to preserve ecosystem integrity (U.S. E.P.A., 2002). Not just agricultural fertilizers cause eutrophication. Storm runoff, landfill leachate, and domestic and industrial effluents also contribute and hindered potential water have the of reuse (Chang et al., 2010).

In addition to nutrients, many heavy metals may occur naturally in groundwater supplies at levels exceeding maximum values for human health. Heavy metals may also be introduced by anthropogenic activities, such as mining, plating, and tanning industries. Globally, abandoned mines have contaminated ground and surface waters and the tanning industry has produced irreversible damage to aquatic environments (Shankar 2008). Chemical spills at industrial sites, improper disposal and storage of wastes, and leaking or abandoned underground storage tanks may all contribute to contamination (USEPA 2002).Studies on urban watersheds in southeast Michigan, United States, for example, showed that current and past land use practices have resulted in groundwater and soil contaminated with chromium, copper, lead, nickel, and zinc (Murray et al. 2006). Hexavalent chromium, in particular, is used in tanning and plating industries, manufacture of dyes and pigments, and in circuit boards. It is on the USEPA list of top twenty total releases and is toxic, carcinogenic, and mutagenic such that the maximum acceptable level in drinking water is 0.1 mgL⁻¹(USEPA 2003). It exists in aqueous solution as either the anion chromate (CrO_4^{2-}) above a pH of 8 and as dichromate $(Cr_2O_7^{2-})$ below a pH

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of 6. At concentrations below 1.6 mgL⁻¹, it may also appear as bichromate $(HCrO_4^{-1})$.

Much work has focused on removal of anions from aqueous solution, usually as single ions, coremoval of nutrients, or co-removal of heavy metals. Recent methods to remove phosphates include adsorption onto a zirconium sulfate surfactant micelle mesostructure (Pitakteeratham et al. 2013), recovery by a continuous struvite reaction crystallization process (Hutnik et al. 2013), immobilization using natural calcium rich sepiolite (Yin et al. 2103), adsorption onto biochar/MgAl-LDH ultra-fine composites (Zhang et al 2013), and enhanced biological removal (Mielczarek et al. 2013).Co-removal of phosphorus and nitrate nutrients has been performed via adsorption onto ceramsite material made from coal ash and metallic iron (Ji et al. 2010), use of plant-microorganism combined systems (Li et al. 2011), and in membrane biological reactors (Galil et al. 2009). Chromium removal has been investigated via integrated membrane bioreactor system (Vyrides and Stuckey 2011), adsorption onto magnetic hydrogel (Yin and Lo 2010), sorption onto animated polyacrylonitrile fibers (Wang et al 2011), adsorption by UV-active functionalized graphene oxide (Dinda et al. 2013), biological reactors (Tekerlekopoulou et al. 2013), and reduction to Cr(III) mediated by zero-valent magnesium (Lee et al. 2013).

In the past decade, the use of naturally occurring or synthetically made clay mineral based lavered double hydroxides. LDHs. has been widely investigated as an inexpensive ion exchange media for removal of anions from aqueous solution. This class of materials has become increasing researched and utilized due to ease of synthesis and low cost compared to resin based ion exchange media. Phosphorus and nitrate removal via super paramagnetic particles modified with LDH was performed by Mandel et al. (2012). Removal of Cr(VI), zinc, and cadmium in a multiion solution (Terry et al. 2010) and removal of nutrients by hydrotalcite, a synthetically made aluminummagnesium based double laver hydroxide. Terry (2009) have been characterized. Phosphorus sorption and recovery using mineral based materials has been investigated by Wending et al. (2006) and Reitzel et al. (2013) have investigated phosphate removal by modified bentonite clay.

This work furthers previous work by Terry et al. (2009, 2010) and investigates the effect of nitrates, phosphates and chromates on the removal of phosphates and chromates via ion exchange with calcined hydrotalcite, HTC in a multi-ion solution. These three were chosen because of the global concern for nutrient contamination of water and the growing concern that industrialization moving into rural and suburban areas will possibly add industrial waste contamination to the same surface and ground water sources. Because it is toxic, carcinogenic, and mutagenic at very low levels, industrial releases of and water contamination by chromium (VI) is of particular concern. Nitrate removal is not considered in this work because previous work has shown that, while nitrates are removed via ion exchange with hydrotalcite in a single ion solution, removal is significantly diminished in the presence of even low phosphate levels. Phosphate removal, on the other hand, is high in a single ion solution and remains so even when up to 30 mgL⁻¹ nitrate is present.

In this study, first the two ion phosphatechromate system is characterized including both the effect of chromate on phosphate removal and the effect of phosphate on chromate removal. Next the three ion system is investigated including the effect of chromate and nitrate on phosphate removal and the effect of phosphate and nitrate on chromate removal. In all experiments, concentrations were used that correspond with those typically found in contaminated surface and ground waters, ranging from zero to 6.13 mgL⁻¹ phosphate (2 mgL⁻¹ P), 30 mgL⁻¹ nitrate, and 20 mgL⁻¹ chromate.

II. EXPERIMENTAL PROTOCOLS

Hydrotalcite, $[Mg_{2}AI(OH)_{6}]_{2}CO_{3}3H_{2}O_{3}$ ion exchange media used in these experiments was purchased from Sigma-Aldrich chemicals. It was calcined by heating at 450°C for several hours. Nitrates were added as sodium nitrate, NaNO₃, phosphates as sodium dihydrogen phosphate, NaH₂PO₄, and chromium (VI) as chromium trioxide anhydrous, CrO₃, all research grade from Fisher Chemicals. Nitrate and phosphate levels were measured using a calibrated Shimadzu UV-1601, UV-visible spectrophotometer. An ascorbic acid soluble reactive phosphorus procedure, HACH standard procedure 8048, using HACH Phosver 3 reagent powder was used for phosphate analysis. The test measurement range was 0 to 0.82 mgL⁻¹ P, reported as 0 to 2.52 mgL⁻¹ PO₄³⁻. Chromium (VI) was measured using a Perkin-Elmer 2280 atomic absorbance spectrophotometer.

All experiments were performed in triplicate in batch mode with 100 ml samples in acid washed high density polyethylene bottles. Samples were prepared by adding the appropriate quantity of nitrates, phosphates and chromate, then adding the hydrotalcite. The alkaline nature of the hydrotalcite caused a pH shift from that of deionized water to about 8.2 + 0.2.Hydrotalcite, HTC, 0.2 gL⁻¹, was added to each solution, which were then shaken for twenty minutes with a NBS gyratory shaker on high speed. Following ion exchange, the HTC was separated from suspension by vacuum filtration though Whatman 0.7 um glass fiber filters and residual concentrations in the aqueous solution were analyzed.

First, the binary system of chromatephosphates was characterized. Following the binary system analysis, the ternary system was characterized. All experiments were performed with phosphate ranges between 0 and 6.13 mgL⁻¹ phosphate (1 to 2 mgL⁻¹ P), 0 to 30 mgL⁻¹ nitrate, and 0 to 20 mgL⁻¹ Cr(VI). Both Freundlich and Langmiur isotherms, typically used to model both ion exchange and adsorption processes, were used to characterize the ion exchange effectiveness, assessing by comparison the effect of other anions in a binary or ternary system on ion exchange removal of the target anion. The Freundlich isotherm,

$$Y = CX_{eq}^{m}$$
,

Where Y = mg of target anion exchanged per unit mass (g) hydrotalcite,

 $X_{\rm eq}$ = residual target anion in solution following ion exchange (mgL^-1), and

C, m = Freundlich model constants is typically used for dilute solutions.

The Langmuir isotherm model

$$Y = bX_{eq}/(1 + KX_{eq})$$

Where Y = mg of target anion exchanged per unit mass (g) hydrotalcite,

 $X_{\rm eq}$ = residual target anion in solution following ion exchange (mgL^-1), and

b, K = Langmiur model constants is more often used and a system is considered favorable when $KX_{\text{eq}}\!>$ 1.

In addition, fixed effects statistical analysis of variance, PROC GLM (SAS Version 9) was used to analyze main and interactive effects for target anion removal and Least Significance Difference t-test and Tukey's studentized range honest significant difference at α =0.05 were used a multiple comparison procedures to determine significant mean difference in treatments. This was performed to analyze any main and interactive effects of, first, phosphate and chromate concentrations on phosphate removal and, second, phosphate and chromate concentrations on chromate removal. The ternary system analysis investigated any main and interactive effects of phosphate, chromate, and nitrate concentrations on first phosphateand then chromate removal.

III. Results and Discussion

Results are presented in the form of residual concentrations and isotherms for the target species. For each isotherm, constants for both Freundlich and Langmiur models are presented with R² values showing strength of fit.Also, as described above, fixed effects analysis of variance was used to determine the effect of the target species and non-target species on removal of the target species.

a) Binary System

First, the effect of Cr(VI) on ion exchange removal of phosphate was measured for low, 0.613 mgL⁻¹, and high, 6.13 mgL⁻¹, phosphate initial concentrations. Cr(VI) ranged from zero to 20 mgL⁻¹. Figure 1 shows residual phosphate as a function of Cr(VI) for both high and low phosphate and Figure 2 gives the comparative isotherms. Langmuir and Freundlich model constants are found in Table 1. Figure 1 shows that at phosphate levels of 0.613 mgL⁻¹, addition of even 1 mgL⁻¹Cr (VI) resulted in a slightly diminished phosphate removal, from 99.5% to 95.2%, respectively. Increasing Cr (CI) concentration caused a continued reduction of phosphate removal to only 88.9% for 20 mgL⁻¹ Cr(VI). For an initial phosphate of 6.13 mgL⁻¹, the presence of Cr(VI) above 2 mgL⁻¹ affected phosphate residuals, but removal remained high. Percent phosphate removal was 99.5% when no Cr(VI) was present and remained above 98.5% up to 15 mgL⁻¹ Cr(VI). Only at 20 mgL⁻¹Cr (VI) did phosphate removal decrease to a still high 96.9%. The three isotherms, each measured over a range of zero to 6.13 mgL⁻¹ phosphate, in Figure 2 verify the result. These were obtained at both low, 2 mgL⁻¹Cr (VI), and high, 20 mgL⁻¹ (Cr (VI), and compared to the isotherm for phosphate alone. The isotherms for just phosphate and phosphate with low Cr (VI) show that low Cr (VI) concentrations did not significantly affect phosphate removal. The isotherm for phosphate in the presence of high Cr (VI), on the other hand, showed that higher Cr (VI) levels do impair phosphate removal.

nodel parameters for the effect of Cr(VI) on phospha									
Freundlich Langmuir									
	m	R ²	b	K	R ²				
	0.7868	0.9996	665.6	7.395	0.9990				
	0.8352	0.9993	578.2	4.987	0.9997				
	0.8123	0.9985	451.7	5.615	0.9991				

Table 1 : Freundlich and Langmuir model parameters for the effect of Cr(VI) on phosphate ion exchange

Fixed effects analysis of variance was used to analyze the effects of phosphate and Cr(VI) on phosphate removal and the Least Significant Difference t-test and Tukey's studentized range honest significant

Cr (VI) mg L⁻¹

0

20

C 253.2

279

197.6

difference at $\alpha = 0.05$ were used as multiple comparison procedures to determine significant mean difference in treatments. The main effect from phosphate was significant (p < 0.02) as was the main Year

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effect from Cr(VI) (p < 0.04), but to a lesser degree, especially for low Cr(VI). The interaction between phosphate and Cr(VI) was also significant (p < 0.03), but again less so than the phosphate main effect because only higher Cr(VI) levels affected phosphate removal significantly.

Next the effect of phosphate on the removal of Cr (VI) was measured for low, 2 mgL⁻¹, and high, 20 mgL⁻¹, Cr (VI) with phosphate varied from zero to 6.13 mgL⁻¹. Figure 3 has residual Cr (VI) as a function of phosphate and Figure 4 shows the Cr (VI) isotherms with low, 0.613 mgL⁻¹, and high, 6.13 mgL⁻¹, phosphate compared to single ion Cr (VI) ion exchange isotherm. Both Freundlich and Langmuir model constants are presented in Table 2.Figure 3 shows that for both 2 mgL⁻¹ and 20 mgL⁻¹ initial Cr (VI), Cr (VI) removal via ion exchange is significantly reduced compared to

single ion removal by the presence of even low phosphate levels. Ion exchange removal of Cr (VI) in a single ion solution was 95% for both high and low initial Cr (VI), but in a binary solution with 0.31 mgL⁻¹ phosphate, removal of 2 mgL⁻¹ Cr(VI) was reduced to 63.5% and that of 20 mgL⁻¹ Cr(VI) was reduced to 69%. For 2 mgL⁻¹ initial Cr(VI), percent removal decreased to about 54% at 0.613 mgL⁻¹ phosphate and remained at that level for higher phosphate concentrations. At 20 mgL⁻¹ initial Cr (VI), removal continued to decrease to only 37% at an initial phosphate concentration of 6.13 mgL⁻¹. The isotherm in Figure 4 also verifies this result. At even low phosphate levels, Cr (VI) ion exchange was reduced greatly across the spectrum of Cr(VI) initial values. This effect was magnified at higher phosphate levels.

PO ₄ ³⁻		Freundlich Langmuir				
mg L ⁻¹	С	m	R ²	b	K	R ²
0	28.28	0.5366	0.9783	34.54	0.2999	0.9983
0.613	15.84	0.499	0.9478	17.18	0.257	0.9858
6.13	8.194	0.6626	0.9795	7.675	0.1075	0.9936

Table 2 : Freundlich and Langmuir model parameters for the effect of phosphate on Cr(VI) ion exchange

Fixed effects analysis of variance, the Least Significant Difference t-test, and Tukey's studentized range honest significant difference at $\alpha = 0.05$ showed the main effect from Cr(VI) was significant (p < 0.02) as was the main effect from phosphate (p < 0.01). The interaction between phosphate and Cr(VI) was also significant (p < 0.01), again showing that both Cr(VI) and phosphate initial concentrations significantly reduce Cr(VI) removal.

b) Ternary System

Once the effects of phosphate and Cr (VI) on each one's respective removal in a binary system was characterized, the ternary system of phosphate, nitrate and chromate was investigated. First, the interactive effect of nitrate, phosphate and Cr (VI) on phosphate removal was determined, then the effect of nitrate, phosphate, and Cr (VI) on Cr (VI) removal was tested. Nitrate removal, as stated, was not considered because previous experiments showed that even low levels of phosphate result in significantly reduced nitrate removal. Nitrate was included in the ternary systems, however, because it is usually present in ground and surface waters contaminated with phosphate and, therefore, may participate in interactive effects.

Figure 5 shows residual phosphate as a function of initial phosphate, Cr(VI), and nitrates. Initial phosphate concentrations ranged between zero and 6.13 mgL⁻¹. Low and high Cr(VI) levels were 2 mgL⁻¹ and 20 mgL⁻¹ and low and high nitrates were 3 mgL⁻¹ and 30 mgL⁻¹. As the figure displays, phosphate removal was

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affected by the presence of both Cr (VI) and nitrate, with the effect increasing at higher initial phosphate levels. There was no significant difference between the reduction in phosphate removal when both low Cr (VI) and either low or high nitrates were present, leading to the observation that nitrates, when Cr (VI) was low did not significantly affect phosphate removal. In both cases, phosphate removal decreased from over 99% for the single ion phosphate system to a minimum of 93.1% for 6.13 mgL⁻¹ initial phosphate, 2 mgL⁻¹Cr (VI) and both 3 and 30 mgL⁻¹ nitrate. As would be expected from the binary system results, for the ternary system of phosphate with 20 mgL⁻¹ Cr(VI) and both 3 mgL⁻¹ and 30 mgL⁻¹ nitrate, there was a greater reduction in phosphate removal. This effect was amplified at 30 mgL⁻¹ nitrate compared to 3 mgL⁻¹ nitrate, showing a greater interactive effect. For the high Cr (VI), low nitrate system, phosphate removal decreased from 95.2% to 85.2% with increasing initial phosphate, while for the high Cr (VI), high nitrate system, it decreased from 92.3% to 75% with increasing initial phosphate. The isotherms for this system, shown in Figure 6, and the isotherm model constants in Table 3 confirm this result. Greatest phosphate removal occurred in the single ion system, after which it decreased a small amount with addition of 2 mgL⁻¹Cr (VI) at both high and low nitrate levels. For these latter two, the isotherms are identical. Declining isotherms for high Cr (VI) and either low or high nitrate show the worst phosphate removal was observed at both high Cr (VI) and high nitrate.

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Cr (v1)	NO₃ ⁻		Freundlich	I		Langmui	r
mg L ⁻¹	mg L ⁻¹	С	m	R^2	b	К	R^2
0	0	253.2	0.7868	0.9996	665.6	7.395	0.9990
2	3	255.9	0.8096	0.9981	601.2	6.22	0.9996
2	30	259.8	0.8145	0.9981	599.2	6.138	0.9998
20	3	243.9	0.9044	0.9992	359.0	1.997	0.9985
20	30	260.8	0.9849	0.9999	277.1	0.286	0.9999

Table 3 : Freundlich and Langmuir model parameters for the effect of Cr(v1) and nitrate on phosphate ion exchange

Fixed effects analysis of variance, the Least Significant Difference t-test, and Tukey's studentized range honest significant difference at $\alpha = 0.05$ showed the main effect from Cr(VI) was significant (p < 0.02) as was the main effect from phosphate (p < 0.01), but the main effect from nitrate was not significant (p < 0.08). As was observed in the binary system, the interaction between phosphate and Cr (VI) was also significant (p < 0.01), again showing that both Cr (VI) and phosphate initial concentrations significantly reduce phosphate removal. The interaction between Cr(VI) and nitrate was also significant (p < 0.03), as is observed by a reduction in phosphate removal between the high Cr (VI) and low nitrate system compared to the high Cr (VI) and high nitrate system. Finally, the interaction between Cr (VI), nitrate and phosphate was determined to be significant (p < 0.03) as would be expected from the binary interactions.

Figure 7 shows residual Cr (VI) as a function of initial phosphate, Cr (VI), and nitrates. Initial Cr (VI) concentrations ranged between zero and 20 mgL⁻¹. Low and high phosphate levels were 0.613 mgL⁻¹ and 6.13 mgL⁻¹ and low and high nitrates were 3 mgL⁻¹ and 30

mgL⁻¹. As the figure displays, Cr (VI) removal was affected by the presence of both phosphate and nitrate, with the effect increasing at higher initial Cr (VI) levels. In a single ion system, Cr (VI) removal was above 95% for all initial Cr (VI) concentrations. As with the binary Cr (VI) and phosphate system, even low phosphate levels reduced Cr (VI) removal and this effect was amplified at higher phosphate levels. For the low phosphate and low nitrate system, Cr (VI) removal fell from a maximum of 85% at 1 mgL⁻¹ initial Cr (VI) to only 46% for 20 mgL⁻¹ initial Cr (VI) and for the high phosphate-low nitrate system, this was reduced further to only 60% and 35% for initial Cr (VI) concentrations of 1 and 20 mgL⁻¹, respectively. The presence of nitrate also interacted to reduce Cr (VI) ion exchange. For the high phosphatehigh nitrate system, Cr (VI) removal fell from only 40% for 1 mgL⁻¹ initial Cr (VI) to 20% for 20 mgL⁻¹ initial Cr (VI). The isotherms, presented in Figure 8 and the model constants, given in Table 4, reinforce this result. The isotherm for the single ion system shows significantly greater removal than the isotherms for any of the ternary systems. Increasing either phosphate or nitrate results in a diminished isotherm.

PO ₄ ³⁻	NO₃⁻		Freundlich			Langmuir	
mg L ⁻¹	mg L ⁻¹	С	m	R ²	b	K	R ²
0	0	28.6	0.5313	0.9709	35.52	0.3098	0.9946
0.613	3	14.7	0.533	0.9613	15.86	0.229	0.9904
0.613	3	14.58	0.536	0.9615	15.69	0.226	0.9903
6.13	30	7.689	0.6786	0.981	7.12	0.0969	0.9936
6.13	30	6.371	0.6763	0.9651	5.714	0.0882	0.9832

Table 4 : Freundlich and Langmuir model parameters for the effect of phosphate and nitrate on Cr (v1) ion exchange

Fixed effects analysis of variance, the Least Significant Difference t-test, and Tukey's studentized range honest significant difference at $\alpha = 0.05$ showed the main effect from Cr(VI) was significant (p < 0.01) as was the main effect from phosphate (p < 0.01)and the main effect from nitrate (p < 0.02). As was observed in the binary system, the interaction between phosphate and Cr (VI) was also significant (p < 0.01), again

showing that both Cr (VI) and phosphate initial concentrations significantly reduce Cr (VI) removal. The interaction between Cr (VI) and nitrate was also significant (p < 0.02), as is observed by a reduction in Cr (VI) removal between the high phosphate-low nitrate system compared to the high phosphate-high nitrate system. Also, the interaction between phosphate and nitrate was significant, resulting in the lowest Cr (VI)

removal under high phosphate-high nitrate conditions. Finally, the interaction between Cr(VI), nitrate and phosphate was determined to be significant (p < 0.02) as would be expected from the binary interactions and the lowest measured Cr(VI) removal at high initial concentrations of all three.

IV. Summary

Experiments were performed to determine the effect of nitrates, chromates (Cr (VI)), and phosphates on the removal of either chromates or phosphates in multi-ion solutions via ion exchange with calcined hydrotalcite, a synthetically made double laver hydroxide. Levels of each anion associated with contaminated ground or surface waters were considered. Both Langmiur and Freundlich isotherm models were applied to characterize and compare each system. Fixed effects analysis of variance was used to analyze both main and interactive effects and the Least Significant Difference t-test and Tukey's studentized range honest significant difference at $\alpha = 0.05$ were used as multiple comparison procedures to determine significant mean difference in treatments.

First, the binary chromate-phosphate system was characterized. It was found that while low Cr (VI) concentrations did not significantly affect phosphate removal, higher Cr (VI) levels did have a negative impact, especially at higher phosphate initial concentrations. The main effects for initial phosphate and Cr (VI) and the phosphate-Cr (VI) interaction were all found to be significant ($\alpha < 0.05$). On the other hand, Cr (VI) ion exchange was significantly reduced by phosphate at both high and low phosphate levels. Both the main effect for Cr (VI) initial concentration and phosphate were significant as was the interaction between the two. At higher Cr (VI) levels, the negative effect of phosphate became intensified.

Characterization of the ternary system showed that the main effects for phosphate initial concentration and Cr(VI) did affect phosphate removal, while the nitrate main effect did not. Isotherms show that phosphate exchange did decrease with increasing Cr (VI), but the effect was greater at higher Cr (VI) initial concentration. The interactions between Cr (VI)phosphate and Cr (VI)-nitrate were also significant, as can be observed by a lowered isotherm capacity as Cr (VI) and nitrates both increased. The lowest phosphate removal isotherm capacity was recorded for the high nitrate-high Cr (VI) case. The ternary interaction was also significant. Finally, Cr (VI) ion exchange was negatively impacted by the presence of phosphates and nitrates to a greater degree than phosphates were affected in the ternary system. The main effects of Cr (VI), phosphate and nitrate and the interactive effects of Cr (VI)phosphate, Cr (VI)-nitrate, nitrate-phosphate, and Cr (VI)-phosphate-nitrate were all significant. Even small

As industrial effluents begin to be introduced into rural areas that already experience nutrient contamination of surface and ground waters, removal of anions in aqueous solution via ion exchange should consider the effect of non-target anions competing for exchange sites with the target anion. Presence of even small amounts of some competing species may significantly reduce the effectiveness of removing target contaminants. In these cases, if it is desired to remove all contaminants, multi-step separation processes removing the most competitive anions first and the least competitive anion last may be required.

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Figure 1 : Residual phosphate as function of Cr(VI)

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Figure 2 : Phosphate ion exchange isotherms as a function of Cr(VI)



Figure 3 : Residual Cr(VI) as a function of phosphate



Figure 4 : Cr(VI) ion exchange isotherms as a function of phosphate



Figure 5 : Residual phosphate as a function of Cr(VI) and nitrate



Figure 6: Phosphate ion exchange isotherms as a function of Cr(VI) and nitrate



Figure 7: Residual Cr(VI) as a function of phosphate and nitrate



Figure 8 : Cr(VI) ion exchange isotherms as a function of phosphate and nitrate



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Streptoquin and Septazole: Antibiotic Drugs as Corrosion Inhibitors for Copper in Aqueous Solutions

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Keywords: copper; HCL; streptoquin and septazole drugs; corrosion inhibition.

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Streptoquin and Septazole: Antibiotic Drugs as Corrosion Inhibitors for Copper in Aqueous Solutions

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Abstract- The inhibiting effect of streptoquin and septazole towards the corrosion of the copper in 0.1M of HCl solution has been studied by potentiodynamic polarization measurements, electrochemical impedance spectroscopy (EIS) and Electrochemical frequency modulation technique (EFM). Potentiodynamic polarization curves indicted that these compounds behave as mixed-type inhibitors. These drugs were adsorbed on copper surface follow Langmuir adsorption isotherm. The results obtained from EIS and EFM are in good agreement with potentiodynamic polarization measurements. Some guantum chemical parameters were calculated and compared with the experimental data.

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I. INTRODUCTION

cid solutions are widely used in different industries. The most important fields of application are acid pickling, industrial acid cleaning, acid descaling [1]. copper is a metal that has been widely used in different types of industries, including electronics due to its relatively high corrosion resistance. Its protection against corrosion saves large costs on repairs and equipment replacements. In spite of copper being a relatively noble metal, it reacts easily in a medium containing oxygen. It has long been known that when copper corrodes in aerated, chloride containing solutions, a layer of Cu₂O is formed on the surface of the metal. In addition, copper is dissolved by reactions which can be summarized as follows [2] :

Anodic:
$$Cu - --- Cu^+ + e$$
 (1)

Cathodic :
$$\frac{1}{2}O_2 + 2H^+ + 2e^- - H_2O$$
 (2)

Then in solution:

$$2Cu^{+} + \frac{1}{2}O_2 + 2H^{+} \longrightarrow 2Cu^{+} + H_2O$$
 (3)

At chloride concentrations lower than 1 M, the copper dissolution occurs through the formation of CuCl, which is not protective enough and is converted to soluble $CuCl_2$ by reacting with excess chloride [3]. According to Bacarella and Griess [4], the anodic dissolution of copper is under mixed control by the

electrodissolution of copper and the diffusion of soluble CuCl² from the Helmholtz plane into the bulk solution. On the other hand-higher chloride complexes such as CuCl₃²⁻ and CuCl₄³⁻ are formed in addition to CuCl and CuCl₂ at chloride concentrations higher than 1M [5]. One of the most important methods in the protection of copper against corrosion is the use of organic inhibitors. Organic compound containing polar groups including nitrogen, sulfur and oxygen[6-18] have been reported to inhibit copper corrosion. The inhibiting action of these organic compounds is usually attributed to their interactions with the copper surface via their adsorption. Polar functions groups are regarded as the reaction center that stabilizes the adsorption process[19]. In general, the adsorption of an inhibitor on a metal surface depends on the nature and the surface charge of the metal, the adsorption mode, its chemical structure and the type of the electrolyte solution[20]. Also, heterocyclic compounds containing nitrogen are known to be good inhibitors of copper and alloys [21]. As the awareness of environment protection, the research is oriented to the development of environmental-friendly corrosion inhibitors[22-25]. Seeking for alternatives of toxic inhibitors is under consideration. Some studies used drugs as green corrosion inhibitors for various metals and alloys[26-30]. The use of environmental friendly pharmaceutical compounds (streptoguin and septazole) as corrosion inhibitors for copper has not been reported before. So, our aim is to study the inhibiting effect of these drugs on copper corrosion in HCl solution using various electrochemical techniques.

II. Experimental

a) Materials and Solutions

Copper was used as the metal electrode for corrosion studies and its chemical composition As follows (weight %): Ni 0.54, Fe 0.066 other impurities 0.76% and Cu 98.04 %. The investigated compounds are belonged to the sulfa group (streptoquin & septazole). As shown in Table 1. These drugs were obtained from Glaxo SmithKline, Medical Union Pharmaceuticals, Alexandria Co. for Pharmaceuticals, Egypt. Analytical grade HCI (37 %) was used as corrosive solution. Double distilled water was used throughout experiments for the preparation of solutions.

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Table 1 : Names, structural and molecular formulas and molecular weights for of the investigated drugs

Names	Structures,	Mol.formulas & Mol. Wt.
Streptoquin Benzenesulfonamide, 4-amino-N-2-thiazolyl N1-2-Thiazolylsulfanilamide	H ₂ N S N H	C ₉ H ₉ N ₃ O ₂ S ₂ 255.32
Septazole M1-(5-methyl-3-isoxazolyl) sulfanilamide		C ₁₀ H ₁₁ N ₃ O ₃ S 268.28

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b) Methods and Techniques

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 disc cut from copper with dimension 1x1 cm which embedded in epoxy with resin of polytetrafluoroethylene. Before measurement, the exposed surface was wetpolished with silicon carbide abrasive paper (800, 1000, 1200 grit), then the electrode was immersed in solution at natural potential until a steady state was reached. All experiments were carried out in freshly prepared solutions and results were always repeated at least three times to check the reproducibility.

Polarization measurements performed at potentials in the range from -450 mV to -150 mV (SCE) at a scan rate of 0.33 mVs⁻¹. All measurements were carried out in aerated solution of 0.1 M of HCl in the absence and presence of different concentrations of inhibitors. The Tafel plots of the anodic and cathodic curves were extrapolated to obtain the corrosion potential ($E_{corr.}$) and corrosion current density ($i_{corr.}$).

Electrochemical impedance spectroscopy were performed at corrosion potentials, E_{corr} , over a frequency range of 10⁵ Hz to 0.5 Hz with a signal amplitude perturbation of 10 mV, using potentiostat/galvanostat (Gamry PCI 300/4) and personal computer with EIS300 software for calculations. Data were presented as Nyqusit and Bode plots. Experiments were always repeated at least three times.

The electrochemical frequency modulation technique (EFM) provides a new tool for electrochemical corrosion monitoring. EFM experiments were performed with applying potential perturbation signal with amplitude 10 mV with two sine waves of 2 and 5 Hz. The choice for the frequencies of 2 and 5 Hz was based on three arguments [31]. 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 CF2 and CF3 [32&33].

measurements All electrochemical were performed using Gamry Instrument (PCI 300/4) Potentiostat / Galvanostat /ZRA. This includes a Gamry framework system based on the ESA 400. Gamry applications include DC105 software for potentiodynamic polarization measurements, EIS300 software for electrochemical impedance spectroscopy and EFM 140 software for electrochemical frequency modulation measurements along with a computer for collecting data. Echem Analyst 6.03 software was used for plotting, graphing, and fitting data.

III. Results and Discussion

a) Potentiodynamic Polarization Measurements

Potentiodynamic polarization curves of copper electrode in 0.1 M HCl solution without and with various concentrations (100, 300, 500, 700, 900 ppm) of streptoquin and septazole at 25°C were recorded. The polarization curves of copper in 0.1 M HCl at 25°C in the presence and absence of different concentrations of streptoquin and septazole are shown in Figs.1 & 2. Electrochemical parameters such as corrosion current density (i_{corr}), corrosion potential (E_{corr}), anodic (β_a), cathodic (β_c) Tafel slopes and inhibition efficiency (η %) were calculated and are listed in Table 2. The inhibition efficiency (η %) and the corrosion rate ($R_{corr.}$) were calculated using Eqs.1&2:

$$\eta (\%) = (i_{\text{corr.}} - i_{\text{corr.}(\text{inh.})} / i_{\text{corr}}) \times 100$$
(1)

$$R_{corr.}(mm/year) = 3270x(i_{corr x EW/D})$$
(2)

where $i_{corr.}$ is the corrosion current density (A cm⁻²), EW the equivalent weight of copper (g) and D the density of Cu (g cm⁻³).



Figure 1 : Potentiodynamic polarization curves of copper in 0.1 M HCl at different concentrations of streptoquin at 25°C



Figure 2 : Potentiodynamic polarization curves of copper in 0.1 M HCl at different concentrations of septazole at 25°C

The presence of increasing amounts of septazole and streptoquin led to a decrease in both the cathodic and anodic current density. It is clear from these data that the inhibitors may affect either the anodic or the cathodic reaction, or both [34]. Since the

anodic (β_a) and cathodic (β_c) Tafel slopes of streptoquin and septazole were found to change slightly with inhibitor concentration, this indicates that these inhibitors affected both of these reactions i.e. these drugs act as mixed type inhibitors [35].

 Table 3 : Electrochemical parameters obtained from potentiodynamic polarization measurements of copper in 0.1 M

 HCl in the absence and presence of different concentrations of streptoquine and septazole at 25°C

Comp	Conc. Ppm	-E _{corr} mV vs SCE	i _{corr} μA cm ⁻²	β _{c,} mV dec ⁻¹	β _a mV dec ⁻¹	R _₽ Ohm cm²	θ	η%	R _{corr.} mmy ⁻¹
	Blank	144	284.10	670	351	352			3.294
	100	154	101.80	650	258	786	0.642	64.2	1.181
	300	180	77.68	708	258	1058	0.727	72.7	0.901
Streptoquin	500	195	61.63	639	234	1206	0.783	78.3	0.715
	700	215	54.29	568	231	1313	0.809	80.9	0.630
	900	212	47.93	501	233	1442	0.831	83.1	0.556
	100	153	53.84	985	247	1590	0.813	81.3	0.624
	300	164	43.58	936	246	1942	0.847	84.7	0.505
Septazole	500	178	34.58	765	237	2296	0.878	87.8	0.401
	700	187	32.55	713	232	2362	0.886	88.6	0.378
	900	197	24.66	566	225	2840	0.913	91.3	0.286

The addition of streptoquine and septazole shift the E_{corr} values towards the negative. A compound can be classified as an anodic-or cathodic-type inhibitor when the change in E_{corr} value is larger than 85 mV [36]. Since the largest displacement exhibited by streptoquine and septazole was about 71 mV as shown in Table 3, it may be concluded that these molecules should considered as a mixed-type inhibitor, meaning that the addition of these drugs to 0.1 M HCl solution both reduces the anodic dissolution of copper and also retards the cathodic hydrogen evolution reactions. From the calculated values of $(\eta\%)$ as shown in Table 3, the order of decreasing inhibition efficiency is as follows: streptoquin > septazole.

b) Effect of Temperature

From Table 4 we found that the corrosion rate increases with the rise of temperature and the inhibition

efficiency decreases with increasing temperature. This can be attributed to the shift of the adsorption-desorption equilibrium towards desorption. Such behaviour suggests that investigated compounds were physically adsorbed on copper surface. The activation energy (E_a^*) of the corrosion process was calculated using Arrhenius eq. (3):

$$i_{corr} = A \exp(-E_a^*/RT)$$
 (3)

Where A is Arrhenius constant, R is the gas constant and T is the absolute temperature. Figures (3&4) show the Arrhenius plot (log i_{corr} vs. 1/T) in the presence and absence of streptoquin and septazole. The values of activation energies E_a^* can be obtained from the slopes of the straight lines and are given in Table (5).

Table 4 : Electrochemical parameters obtained from potentiodynamic polarization measurements of copper in 0.1 M
HCI in presence of 500 ppm from streptoquine and septazole at different temperature

Comp	TempK	-E _{corr} mV vs SCE	i _{corr} µA cm⁻²	β _{c,} mV dec ⁻¹	β _{a,} mV dec ⁻¹	R _₽ Ohm cm²	θ	η%	R _{corr.} mmy ⁻¹
Streptoquin	000	173	99.16	578	269	803	0.736	73.6	1.150
Septazole	308	119	58.16	516	217	1140	0.846	84.6	0.675
Streptoquin	318	148	128.6	600	239	577	0.671	67.1	1.491
Septazole		166	103.1	606	252	749	0.736	73.6	1.196
Streptoquin	000	182	154.00	499	151	517	0.636	63.6	1.786
Septazole	328	165	127.00	430	185	579	0.700	70.0	1.473


Figure 3 : Log i_{corr} vs. (1/T) curves for the corrosion of copper in 0.1 M HCl in the absence and presence of different concentrations of streptoquine at different temperatures



Figure 4 : log (i_{corr}/T) vs. (1/T) curves for the corrosion of copper in 0.1 M HCl in the absence and presence of different concentrations of streptoquine at different temperatures

It is noted that the values of activation energy is higher in the presence of drugs than in their absence indicating that inhibitors exhibit low η % at elevated temperatures [37] and also, due to the film formation on copper surface. An alternative formulation of the Arrhenius equation is the transition state equation [38]:

$$i_{corr} = RT/Nh \exp(\Delta S^*/R)\exp(-\Delta H^*/RT)$$
(4)

where h is Planck's constant, N is Avogadro's number, ΔS^* is the entropy of activation and ΔH^* is the enthalpy of activation. Figures)5&6) show a plot of log (i_{corr}/T) vs. (1/T). Straight lines were obtained with slopes

of	(ΔH [*] /2.303R)	and	intercepts	of	(log	R/	Nh
$+\Delta$.S*/2.303R)						

Table 5 : Kinetic-thermodynamic corrosion parameters for copper corrosion in the absence and presence of various concentrations of streptoquin and septazole

Inhibitors	Conc. ppm	E₄* kJ mol⁻¹	-∆H* kJ mol ⁻¹	-∆ S* J mol⁻¹ K⁻¹
	Blank	9.40	19.1	226.3
	100	23.4	22.1	209.7
Strantoquin	300	24.7	23.9	205.5
Streptoquin	500	25.1	24.7	203.6
	700	25.3	25.7	201.7
	900	25.7	26.6	200.4
	100	37.9	22.9	192.4
	300	38.3	24.9	190.5
Septazole	500	39.1	26.8	188.6
	700	40.2	28.7	186.7
	900	40.8	30.6	184.7

The values of ΔH^* and ΔS^* were calculated and listed in Table (5). The negative values of ΔH^* reflect that the process of corrosion is an exothermic one. The negative values of ΔS^* implies that the activation complex is the rate determining step that represents an association rather than dissociation step [39]. This means that the activated molecules are in higher order state than that the initial state.

c) Adsorption Isotherm

In order to get a better understanding of the electrochemical process on the metal surface, adsorption was studied. For determining the related mechanism, adsorption isotherms were drawn. A number of mathematical relationships for the adsorption isotherms have been suggested to fit the experimental data of the present work. The simplest equation is that due to [40] Langmuir which is given by the general relation:

$$C/\Theta = 1/K_{ads} + C$$
 (5)

where K_{ads} is the equilibrium constant for the adsorption/desorption process, C is the inhibitor concentration in the bulk of the solution in mol L⁻¹. From the intercepts of the straight lines on the C/ Θ axis, one can calculate K_{ads} values that relate the standard free energy of adsorption, (ΔG°_{ads}), as given by this Eq. [41]:

 $K_{ads} = 1/55.5 \exp(-\Delta G_{ads}^{\circ}) / RT$ (6)

The value of 55.5 is the concentration of water in the bulk solution in mol L⁻¹. Free energy, ΔG°_{ads} , values were calculated and are given in Table (6), the negative values of ΔG°_{ads} indicate spontaneous adsorption of inhibitors on copper surface [42]. Generally, values of ΔG°_{ads} up to -20 kJ mol⁻¹ are

consistent with physisorption, while those around -40 kJ mol⁻¹ or higher are associated with chemisorptions as a result of the sharing or transfer of electrons from inhibitor molecules to the metal surface to form co-ordinate bond [43]. the calculated ΔG°_{ads} values are 22-36 kJ mol⁻¹ indicating that the adsorption mechanism of septazole and streptoquin in 0.1 M HCl solution at the studied temperatures is mixed one (physisorption and chemisorption). Figs. (7&8) show that the plot of C/ θ vs. C for streptoquin and septazole are linear plots which obtained for the different temperatures and inhibitors, indicating that the adsorption of these compounds followed Langmuir adsorption isotherms. The various adsorption parameters obtained from this isotherm are listed in Table (6).



Figure 7: The linear form of Langmuir adsorption isotherm of streptoquine on copper surface in 0.1 M HCl solution





Thermodynamic adsorption parameters such as enthalpy of adsorption ΔH_{ads}° and entropy of adsorption ΔS_{ads}° can be deduced from integrated version of the Vant't Hoff equation expressed by [44]:

Table 6 : Thermodynamic parameters for the adsorption of streptoquine and septazole on copper in 0.1 M HCl a
different temperature

Inhibitors	Temp., K	K _{ads} x10 ⁻⁴ M ⁻¹	- ∆ G [°] _{ads} kJ mol ⁻¹	- ∆ H [°] _{ads} kJ mol⁻¹	- Δ S° _{ads} J mol ⁻¹ K ⁻¹	
	298	0.72	30.7			
Straptoquip	308	0.53	28.1	01.0	107.6	
Streptoquin	318	0.30	23.4	31.2		
	328	0.25	21.9			
	298	1.44	36.4			
Contorolo	308	1.11	34.3	45.0	147.6	
Septazole	318	0.36	24.9			
	328	0.35	24.4			

$$InK_{ads} = (-\Delta H_{ads}^{\circ}/RT) + (\Delta S_{ads}^{\circ}/R) + In(1/55.5)$$
(7)

Figure 9 shows the plot of In $K_{ads.}$ Vs. 1/T which gives straight lines with slopes (- $\Delta H^{^{\prime}}_{ads}/2.303R)$ and

intercepts [($\Delta S_{ads}^{\circ}/2.303R$) + In (1/55.5)]. Calculated values of ΔH_{ads}° and ΔS_{ads}° using the Van't Hoff equation are listed in Table 6.



Figure 9 : Log K_{ads} vs. (1/T) curves for the corrosion of copper in 0.1 M HCl in the absence and presence of different concentrations of septazole and streptoquine at different temperatures

Negative sign of ΔH°_{ads} indicates that the adsorption of septazole and streptoquine on copper surface is an exothermic process. ΔH°_{ads} is about 31.2-45 kJ mol⁻¹ which is larger than the common physical adsorption heat (40 kJ mol⁻¹), but smaller than the common chemical adsorption heat (100 kJ mol⁻¹) [45], probably meaning that both physical and chemical adsorption take place (i.e. comprehensive adsorption). The negative ΔS°_{ads} values are accompanied with

exothermic adsorption process. This is agrees with the expected, when the adsorption is an exothermic process, it must be accompanied by a decrease in the entropy change and vice versa [46].

d) Electrochemical Impedance Spectroscopy

Electrochemical impedance is a powerful tool in the investigation of the corrosion and adsorption phenomena [47]. The correlation of the experimental impedance plot to an equivalent circuit enables the verification of the mechanistic model for the system. Such a correlation leads to the calculation of the numerical values corresponding to physical and/or chemical properties of the electrochemical system [48, 49]The impedance data of Cu, recorded after 30 min immersion in 0.1 M HCl are presented as Nyquist and Bode plots in presence and absence of various concentrations of streptoquin and septazole are shown in Figures (10&11), respectively. The Nyquist plots show

a single semi-circle shifted along the real impedance (Z_r) axis, single capacitive loop, which is attributed to charge transfer of the corrosion process, and the diameters of the loops increase with the increase in the concentration of the drugs. The Bode plots show resistive region at high frequencies and capacitive region at intermediate frequencies but do not show a clear resistive region (horizontal line and a phase angle = 0) at low frequencies. These plots show two overlapped phase maxima at intermediate and low frequencies.



Figure 10 : The Nyquist (a) and Bode (b) plots for corrosion of copper in 0.1 M HCl in the absence and presence of different concentrations of streptoquin



Figure 11 : The Nyquist (a) and Bode (b) plots for corrosion of copper in 0.1 M HCl in the absence and presence of different concentrations of septazole

The impedance data of the copper electrode in the presence of different inhibitors concentrations were analyzed using the equivalent circuit shown in Figure 12. This circuit includes constant phase elements (CPE) in place of capacitors to represent various types of non homogeneities typical of corroding electrodes such as surface roughness, insufficient polishing, grain boundaries and surface impurities [50]. The impedance of this element is frequency dependent, and can be mathematically expressed using two parameters, Y_o and n as:

$$Z_{CPE} = Y_{o}^{-1}(j \omega)^{n-1}$$
 (8)

where Y_o is the CPE coefficient, ω the angular frequency ($\omega = 2\pi f$, where f is the AC fr According to the value of n, Eq. (8) accounts for an inductance (n = -1), a resistance (n = 0), a Warburg impedance (n = 0.5) or a capacitance (n = 1). A Warburg element

occurs when charge carriers diffuse through a material. A rough or porous surface can cause a double layer capacitance to appear as a constant phase element with n varying between 0.5 and 1. This circuit is typical of oxide-coated metals and has been used before by other authors in relation to copper corrosion in potable water [51]. R_s represents the solution resistance between the electrode surface and the tip of the Luggin capillary, Z_{CPE_0} a constant related to the surface oxide, R_0 the resistance to current flow through defects in the surface oxide, Z_{CPEdI} a constant related to the double layer and R_{dl} the double layer resistance. From these Nyquist plots, the difference in real impedance at lower and higher frequencies is generally considered as chargetransfer resistance. The resistances between the metal and outer Helmholtz plane (OHP) must be equal to the $R_{\rm ct}$. The adsorption of inhibitor molecules on the metal surface decreases its electrical capacity because they displace the water molecules and other ions originally adsorbed on the metal surface. This modification results in an increase of charge-transfer resistance. The $R_{\rm ct}$ values increased with inhibitors concentrations may suggest the formation of a protective layer on the copper surface. This layer makes a barrier for mass and charge-transfer. The values of the charge-transfer resistance (R_{ct}) were obtained from the difference in real component (Z) of impedance at lower frequencies. Also the double layer capacitances (C_{dl}) were calculated by the Eq. (9) [31].

$$C_{cl} = (2\pi f_{max} R_{cl})^{-1}$$
(9)

where f_{max} is the frequency value at which the imaginary component (Z") of impedance is maximum. The calculated values of R_{ct} and C_{dl} are listed in Tables (7). The data obtained from fitted spectra are listed in Table 7.The degree of surface coverage (θ) and $\%\eta$ were calculated from the EIS data using following equation:

 $\%\eta = \theta \times 100 = [1 - (R_{ct}^*/R_{ct})] \times 100$ (10)

where R_{ct} and R_{ct}^* are the charge-transfer resistances with and without the inhibitors, respectively. θ and η are also listed in Table 7, and also show that the R_s values are very small compared to the R_{ct} values. By increasing the inhibitor concentrations, the R_{ct} values increase and the calculated C_{dl} values decrease, which causes an increase of θ and η %, the R_{ct} are obtained by septazole at all concentrations is higher than that obtained by streptoquin. The order of the IE obtained from the R_{ct} values is: septazole > streptoquin.

As it can be seen from Tables 7, the C_{dl} values tend to decrease with the increase of the concentration of the surfactants in 0.1 M HCl solution. The decrease in the C_{dl} , which can result from a decrease in local dielectric constant and/or an increase in the thickness of the electrical double layer, suggests that surfactants molecules function by adsorption at the metal/solution

interface. The inhibition efficiencies calculated from impedance data are closed to those obtained from potentiodynamic polarization measurements. The results show the good agreement between measurements obtained from both techniques.



Figure 12: Equivalent circuit proposed to fit the experimental data when two time constants are present

Table 7: Electrochemical kinetic parameters obtained by EIS technique for copper in 0.1 M HCl in the absence and presence of different concentrations of streptoquin and septazole

Comp.	Conc. ppm	R _p ohm cm²	C _{dl} µFcm⁻²	θ	η%
	Blank	172.7	2640		
	100	193.4	1079	0.107	10.7
Streptoquin	300	246.4	1013	0.299	29.9
	500	268.0	633	0.359	35.9
	700	269.4	520	0.366	36.6
	100	304.1	1980	0.432	43.2
Sontazolo	300	418.8	1910	0.588	58.8
Sepiazole	500	447.4	1590	0.614	61.4
	700	491.9	1560	0.649	64.9

e) Electrochemical Frequency Modulation (EFM)

Electrochemical frequency modulation (EFM) a new tool for monitoring technique is the electrochemical corrosion. EFM is non-destructive technique, rapid test, gives directly values of the corrosion current without a prior knowledge of Tafel constants and has also great strength due to the causality factor, which serve as an internal check on the validity of the EFM measurements Table 8 shows the corrosion kinetic parameters such as inhibition efficiency $(E_{\text{FFM}} \%)$, corrosion current density ($\mu \text{ Acm}^{-2}$), Tafel constants ($\beta_a \& \beta_c$) and causality factors (CF-2, CF-3) at different concentrations of drugs at 25°C. Figures 13-15 represent the EFM intermodulation spectra of copper in 0.1 M HCl devoid of and containing 500 ppm concentrations of streptoquin and septazole, respectively. The inhibition efficiency, E_{FFM}% of streptoquin and septazole was calculated using eq. (1). As can be seen from Table 8, the corrosion current densities decrease with increase in streptoquin and

septazole concentrations. The causality factors indicate that the measured data are of good quality. If the causality factors differ significantly from the theoretical values of 2.0 and 3.0 then it can be deduced that the measurements are influenced by noise. If the causality factors are approximately the predicted values of 2.0 and 3.0, there is a causal relationship between the perturbation signal and the response signal. Then the data are assumed to be reliable [32]. The calculated inhibition efficiency E_{EFM} % enhances with increasing streptoquin and septazole concentration.



Figure 13 : Intermodulation spectra recorded for copper electrode in 0.1 M HCl solutions



Figure 14 : Intermodulation spectra recorded for copper electrode in 0.1 M HCl solutions in the absence and presence 500 ppm of streptoquin



Figure 15 : Intermodulation spectra recorded for copper electrode in 0.1 M HCl solutions in the presence 500 ppm of septazole

Table 8 : Electrochemical kinetic parameters obtained from EFM technique for copper in 0.1 M HCl in the absence and presence of different concentrations of streptoquin and septazole

Comp.	Conc. ppm	i _{corr} µA cm⁻²	β _a mVdec ⁻¹	β _c mVdec ⁻¹	E _{efm} %	CR mpy	CF-2	CF-3
	Blank	135.00	40	57		34.66	1.828	3.845
	100	134.30	42	61	0.52	33.98	2.093	3.361
Streptoquin	300	122.30	37	50	9.4	32.00	1.997	3.781
	500	95.68	37	55	29.13	25.03	2.167	3.250
	700	77.35	28	39	42.70	20.23	1.908	3.938
	100	77.34	40	65	42.70	20.23	1.976	3.843
Septazole	300	57.55	36	59	57.40	12.64	2.001	3.644
	500	49.88	41	67	63.10	13.05	1.942	3.130
	700	48.31	45	81	64.21	15.05	2.062	3.308

f) Quantum Chemical Calculation

Over the past decades the semi empirical molecular orbital methods have been widely used in computational studies. Semi empirical methods serve as efficient computational tools which can yield fast quantitative estimates for a number of properties [52]. MNDO (modified neglect of differential overlap) is a semiempirical methods based on the NDDO (neglect of diatomic differential overlap) approximation. The guantum chemical calculations were employed to give further insight into the mechanism of inhibition action of streptoquin and septazole. For this purpose the highest occupied molecular orbital E_{HOMO} , energy of the lowest unoccupied molecular orbital (E_{IUMO}), energy gap (ΔE) between LUMO and HOMO and Mulliken charges on charge we can observe the presence of excess of negative charge on nitrogen, oxygen and sulfur atoms can be adsorbed on the copper surface using these active centres leading to the corrosion inhibition action.

The calculated parameters such as E_{HOMO} , E_{LUMO} and the dipole moment (μ) of the two inhibitors were shown in Table 9, the dipole moment (μ) is an indicator of the electronic distribution in a 1molecule and is one of the properties used to discuss and to rationalize the structure. Low value of the dipole moment (μ) will favour the accumulation of inhibitor molecules on the metallic surface. The comparison between the calculated dipole moments of inhibitors reveals that the lower septazole has better inhibition efficiency. E_{HOMO} often indicates the electron donating ability of the molecule and the inhibition efficiency increases with increasing E_{HOMO} values. High E_{HOMO} values indicate that the molecule has a tendency to donate electrons to appropriate acceptor molecules with low energy empty molecular orbitals. Increasing of the values of the E_{HOMO} facilitates adsorption (and therefore inhibition) by influencing on the transport process through the adsorbed layer. HOMO and LUMO electronic density distribution of streptoquin and septazole were plotted in Figure 15. There is a general consensus by several authors that the more negatively charged heteroatom, the more it can be adsorbed on the metal surface through donor-acceptor type reaction [53]. From the values of Mulliken charge we can observe the presence of excess of negative charge on nitrogen, oxygen and sulphur atoms can be adsorbed on the copper surface using these active centres leading to the corrosion inhibition action. Table 9 shows the quantum chemical calculation parameters

 $(E_{HOMO}, E_{LUMO}, \Delta E = (E_{LUMO} - E_{HOMO})$, dipole moment (μ), heat of formation and ionization potential) for streptoquin and septazole. From these tables the less negative E_{HOMO} and the smaller ΔE reflect a great inhibition efficiency of the inhibitors [54]. From the energy of highest occupied molecular orbital (E_{HOMO}), the order of inhibiting efficiency is as follow: septazole > streptoquin. This is a good agreement with the experimental data obtained by potentiodynamic polarization, EIS and EFM techniques.

	Streptoquin	Septazole	
НОМО			
LUMO			
Mliken charge	0.094 -0.014 -0.186 -0.187 -0.050 0.046 0.308 -0.139 0.308 -0.139 0.308 -0.139 -0.308	-0.055 -0.055 -0.055 -0.033 -0.129 -0.187 -0.191 -0.191 -0.191 -0.191 -0.191 -0.191 -0.191 -0.191 -0.191	

Figure 15: Molecular orbital plots and Mulliken charge of streptoquin and septazole using MNDO

Table 9 : The calculated quantum chemical parameters	
of streptoquin and septazole	

Parameters	streptoquin	Septazole
E _{HOMO} (ev)	-9.27	-9.19
E _{LUMO} (ev)	-0.71	-0.94
ΔE(ev)	8.56	8.25
Dipole moment (µ) (debye)	6.23	4.44
Surface area Å ²	252.88	263.80

IV. Conclusions

The corrosion resistance of copper increases with the increasing concentration of streptoquin and septazole and decreases with increasing temperature indicating that the inhibition occurs through mainly physical adsorption of the investigated drugs on copper surface. These drugs act as mixed-type inhibitors, suppressing the corrosion reactions by forming a protective adsorption film on copper surface. Adsorption

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of these inhibitors on copper surface obeys Langmuir isotherm. The increase in concentration of the investigated drugs leads to increase in the values of the charge transfer resistance (R_{cl}) and a decrease in the values of double layer capacitance (C_{cl}). The results obtained using these different techniques were in good agreement verifying the validity of data obtained by these techniques. Comparison of quantum chemical calculations and experimental data exhibit good correlation confirming the reliability of the methods employed here.

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Experimental Determination of Bubble Size in Solution of Surfactants of the Bubble Column

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Experimental Determination of Bubble Size in Solution of Surfactants of the Bubble Column

Maedeh Asari ^a & Faramarz Hormozi ^o

Abstract- This paper focuses on the effect of surfactants on the bubble size. Bubble size in SDS/water system were investigated at various superficial gas velocities (0.13, 0.26 and 0.5 cm/s). On the other hands, Bubble diameter were determined for different values of SDS surfactant concentration. Surfactant concentration in water were 0.05, 0.02 and 0.1 vol.%. Tap water and aqueous solutions with surfactants (anionic, non-ionic and zwitterionic) are used as liquid phases. The bubbles size in this phase is determined at $C_s = 0.05\%$ vol and $u_q = 0.13$ cm/s. The bubbles are generated into a small-scale bubble column making of Plexiglas with height of 1.2m. High speed photography techniques are used to measure the bubble size. The experimental results were shown that bubble diameter in SDS/water system is larger than other systems. In solution of SDS, Sauter mean bubble diameter (Location A and D) decreases when superficial gas velocity increased.

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I. INTRODUCTION

ubble column reactors are widely used in chemical and biochemical processes such as oxidation, chlorination, polymerization, hydrogenation, synthetic fuels by gas conversion processes, fermentation and wastewater treatment. Bubble columns can be employed in many mass transfer processes[3]. However, the lack of a more complete knowledge on the bubble column fluid dynamic behavior in its various regimes causes several operational difficulties and design uncertainties, which include poor predictions of the mean bubble diameter, gas hold up and interfacial area[2, 14]. A bubble column reactor is basically a cylindrical vessel with a gas distributor at the bottom [12]. The interfacial area available for mass transfer is the most important design parameter defined by gas holdup and bubble size which in turn are affected by the operating conditions, the physic-chemical properties of the two phases, the gas sparger type and the column geometry [5]. Bubble column are preferred to be two-phase contactors for their ease of operation, maintenance and absence of moving parts, yet they have complex hydrodynamics characteristics [21]. Knut [13] studied dynamic simulation of 2D bubble column and shown that two dimensional dynamic simulation of the flat bubble

column is feasible, applying state-of the art dynamic turbulence models. Surfactant designates a substance that exhibits some superficial or interfacial activity. Different methods have been employed for bubble dimension evaluation [9]. Gas bubbles in transparent fluids can be photographed and measured, usually using image-analysis [16]. This is the simplest technique but cannot be used with opaque media 2 such as those found in fermentation systems. Statistical models [6] are required to calculate bubble -size distributions from the measured chord lengths. Several authors studied bubble size and interfacial phenomena in different types of bubble column reactors. Colella et al.[7] studied the interfacial mechanisms focusing on the coalescence and breakage phenomena of bubbles in three different bubble columns. They investigated the influences of gas superficial velocity and different hydrodynamic configurations on bubble size distribution in the bubble columns. Lehr and Mewes [15] evaluated the bubble sizes in two-phase flows. They predicted the bubble size distribution in bubble columns including the formation of large bubbles at high superficial gas velocities. Schäfer et al. [20] discussed the influence of operating conditions and physical properties of gas and liquid phase on initial and stable bubble sizes in a bubble column reactor under industrial conditions. Akita and Yoshida [1] determined the bubble size distribution using a photographic technique. The gas was sparged through perforated plates and single-orifice using various liquids (water, aqueous and pure glycol, methanol, and carbon tetrachloride). It has been reported in the literature [11] that with increase in surfactant concentration, coalescence time increases. Sardeing et al. [19] reported that in superficial gas m/s, bubble velocities between $1.5 \times 10^{-4} - 2 \times 10^{-4}$ diameter was in surfactant solutions between 1mm-8mm. In these studies we have also analysed the influence of SDS surfactant concentration and the gas flow-rate upon the bubble diameter in bubble column. On the other hand, the bubble size distribution has been studied in ionic, nonionic and zwitterionic surfactants on the bubble column.

II. EXPERIMENTAL SETUP AND TECHNIQUE

The schematic diagram of the modified bubble column is shown in Fig.1. It consists of an air compressor, (1), a rotameter, (2), an halegon lamp, (3), etc. Four different surfactant (Tween20, [CAS

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No:9005-64-5], Triton X 100, [CAS No:9002-93-1], Cocoamidopropy Betaine, [CAS No:61789-40-0], SDS, [CAS No:151-21-3]) have been used in the present work. All of surfactants were purchased from Merck company (Germany). Bubble size is reported at ambient conditions [atmospheric pressure 25 (± 0.5)°C]. The gas from the compressed air line passed through calibrated rotameter. The photographic method, used in this study to determine the bubble size of the two-phase mixture, has been developed using a rectangular bubble column $(20 \text{cm} \times 5 \text{cm} \times 120 \text{cm})$. The liquid column heights during the operation were 45cm. To determine profiles of ellipsoid, bubble was monitored over distance ca. 1m and was using professional video recorder. The photographs were taken by a digital camera (Casio Exilim (EX-F1)) taken along the height of the column, from bottom to top. The digital photographs were processed and enhanced by using Image Processing MATLAB Software that enabled to distinguish clearly the bubble boundaries. The diameters of the bubbles were determined from photographs of the operating column, 5, 20, 30 and 40 cm above the gas distributor. The images were taken at three axial positions for different operating conditions. The 2d picture shapes of the bubbles were approximated by ellipsoid [17, 18] whose maximum and minimum axes were automatically computed by the software program used for image analysis. The third dimension was calculated with the

assumption that the bubbles are symmetric around the minimum axes. From the known values of maximum and minimum axes, an equivalent ellipsoid bubble diameter was calculated by the following equation [8]:

$$\mathbf{d}_{\mathrm{be}} = \sqrt[3]{d^2}_{\mathrm{b,\,max}} d_{\mathrm{b,\,min}} \tag{1}$$

Where $d_{b, max}$ and $d_{b, min}$ are the maximum and minimum bubble diameter of bubble. The distributions were obtained by sorting the equivalent diameters of bubbles into different uniform classes. At a particular operating condition, the bubble picture taken from different locations of the column are shown in Fig. 2. The Sauter mean bubble diameter (d_{vs}) is defined as the volume-to-surface mean bubble diameter [4]:

$$d_{vs} = \frac{\sum_{i=1}^{N} n_i d_{Bi}^3}{\sum_{i=1}^{N} n_i d_{Bi}^2}$$
(2)

Where n_i is the number of bubbles of diameter d_{Bi} .

Between 1000 and 3000 bubbles were counted for determination of the size distribution, using 30 photographs.



Fig. 1 : Gas-liquid experimental set-up. (1): Air compressor;(2): Rotameter; (3): Halogen lamp; (4): Plexiglas plate; (5): Bubble column; (6): Camera; (7): Image processing

III. BUBBLE SIZE DISTRIBUTION

Bubble coalescence and breakup play a significant role in determining bubble size distribution. Coalescence was found to take place when more than about a half of the projected area of the following bubble was overlapped with that of the leading bubble at the critical distance. In contrast, the breakup occurred in the case the overlapping was less than about a half of the projected area of the following bubble. Thus, when the leading bubble is larger than the following one, the latter has a tendency to coalesce. In contrast, in the case of the smaller size of the leading bubble, the following bubble tends to breakup. Coalescence is significantly influenced by the physical properties of the

liquid. Analysis of bubble size in bubble columns must distinguish between bubble-size distribution just after bubble formation at the sparger and size distribution further away from the distributor [17]. Two basic methods – photography and probe techniques – exist for determining bubble size, however; they do not lead to identical results. Both methods are subject to certain limitations in view of the marked bubble selection that may occur (i.e., not all bubble sizes can be detected). In particular, any measurement method only leads to realistic results if the flow is homogeneous (i.e., a narrow bubble-size distribution is found). As yet, no method can be recommended for the measurement of large bubbles in the heterogeneous flow regime.

IV. RESULTS AND DISCUSSION

a) Effect of superficial gas velocity upon bubble size in SDS + water system

First, there is general observation that applies to all solutions. For example, regardless of type and presence of chemical added, the average bubble radius decreases as gas flow rate. Fig. 3 show bubble size distribution for SDS-water system in regions A and D. As the gas flow rate increases the gas holdup and kinetic energy increases which increase turbulent intensity, bubble- bubble interactions, velocity of bubbles and the probability of coalescence which is because of as increasing collision frequency between bubbles with increase in gas flow rate.



Fig 2 : Photograph taken from different location of column in Tween20/water system at C_s =0.05%vol and u_q =0.13cm/s Location A , B , C and D

The probability of coalescence is higher in region D but the bubble size decreases with increasing superficial gas velocity in A and D location. This is due to bubble break- up with increasing gas flow rate. Also as the superficial gas velocity increases, the Sauter mean bubble diameter decreases (Fig. 4). For u_g greater than 0.13 cm/s smallest bubbles are obtained in solution of lowest static surface tension. The rate of coalescence decreases with the gas flow rate increasing.



Fig 3 : Effect of gas flow rate upon bubble size in SDS/water system at C_s =0.02%vol. (a) Location A; (b) Location D





b) Effect of SDS concentration upon bubble size

One of the parameters that effect bubble size, is surfactant concentration. Effect of various SDS concentrations (0.02, 0.05 and 0.1%vol) at u_g =0.13cm/s on bubbles diameter is shown in Figure 5. SDS addition to pour water decreased the bubbles diameter. Further, surfactant concentration enhancement decreased the of bubbles diameters by decreasing the surface tension and buoyancy force. Sardeing et al. [19] used various surfactants and investigated that bubbles diameter decreased about 30% (as an average value). The bubble size distribution in an emulsification processes is

a result of the competition between opposite processes, bubble breakage and bubble-bubble coalescence. It was shown experimentally that the bubble size rapidly decreases with an increase of SDS concentration [10]. Sample photographs of the bubble populations shown in Fig. 6. They clearly showed that as the SDS concentration increases, the bubble populations will become smaller in size. Sauter mean bubble diameter (d_{vs}) decreases due to SDS concentration increasing (Fig.7).



(b)

Fig 5: Influence of SDS concentration upon bubble diameter at u_q =0.13cm/s; (a) Location A. (b) Location D 6



Fig 6 : Example of photograph of bubbles at u_g =0.13cm/s for SDS/water system; (a) C_s=0.02%vol; (b) C_s= 0.05%vol; (c) C_s= 0.1%vol



Fig 7: Effect of SDS concentration upon sauter mean diameter at u_q =0.13cm/s

c) Effect of ionic, non-ionic and zwitterionic surfactants on bubble size

Presence of surfactants has a great effect on the bubble diameters. The bubble size distribution was obtained in four axial locations A (of height 0.05 m), B (of height 0.2 m), C (of height 0.3m) and D (of height 0.4m) from the bottom of the column (Fig. 2). Typical results for these four locations are presented in Fig. 8. It is seen that the bubble size in location D are greater than location A , B and C (Fig. 2). The average bubble size in location C and B are almost the same. All calculations regarding goodness of fit have been performed by MATLAB software. Bubble diameter increased with increasing the distance from the bottom of the column due to the coalescence of smaller bubbles. The coalescence bubbles of location A go up due to their buoyancy and accumulate in location B, C and D. Also the bubble number flux varies in different locations due to the same reason. That bubble number flux decreases in location C and D over location A and B is result of an increase in bubble size due to coalescence. As shown in Fig. 8, there is no significant variation of bubble size in location B and C. The bubble size in location A is much smaller than other locations due to a break- up.



(a)













Fig.8. Bubble size distribution for solution surfactant with water location of (a) h=5cm; (b) h=20cm; (c) h=30cm; (d) h=40cm at C_s =0.05%vol and u_g =0.013cm/s

Fig 8. Shows the relation between the detached bubble diameter and fraction of bubbles for the different surfactants. whatever the liquid properties are, the bubble diameters vary between 0.2 and 0.8 for u_{q} = 0.13cm/s (d<0.2cm, effective force is surface tension and bubbles are spherical). For this gas flow rate, the order below is observed:

$d_{SDS} > d_{Betaine} > d_{Triton X-100} > d_{Tween20}$

Sauter mean bubble diameter was investigated in four different axial positions (A, B, C and D). Typical





Fig 9 : Sauter mean bubble diameter as a function of distance from gas sparger

20

h(cm)

0

×

×

Ö

 \Diamond

Δ

8

w/Sds

30

w/Tween20

w/Betaine

w/Triton X-100

8

40

IV. CONCLUSION

Effect of surfactant on the bubble size in rectangular bubble column has been studied. In order to obtain bubble size distribution about 1000-3000 bubbles were analyzed. The evaluation of bubble size distribution in different location of the column and the influence gas flow rate and SDS concentration were pointed out. The were done usina measurements photographic techniques. The bubble size in bubble column increased with increasing distance from the bottom of the column due to coalescence. The bubble diameter in SDS+ water system were bigger than other system. When gas flow rate increase (SDS +water system), an increase in the number of small bubbles was also observed, and Sauter mean bubble diameter also decreased due to breakage bubbles. The Sauter mean bubble diameter decreases, when SDS concentration increasing.

v. Nomenclature

- $d_{b,max}$ major axis of the projected ellipsoid(m) 9
- $d_{b,min}$ minor axis of the projected ellipsoid(m)
- d_{vs} sautor mean diameter bubble(m)
- *u_g* superficial gas velocity (m/s)
- *h* distance from gas distributor(m)
- d diameter(m)
- C_s surfactant concentration

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Approach:

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References	Complete and correct format, well organized	Beside the point, Incomplete	Wrong format and structuring

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