Experimental and theoretical studies of pressure swing adsorption process to remove H$_2$S from methane in a packed bed

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In this research, an experimental study coupled with a mathematical modeling for a modified pressure swing adsorption (PSA) technique is developed. In particular, solid grains are saturated with a liquid selective solvent of NMP (n-methyl-2-pyrolidone) to remove H$_2$S from a gaseous mixture. Since, the solubility of H$_2$S is very sensitive to the partial pressure above the layer of liquid, the saturated sorbent could be easily regenerated by sweeping its bed with nitrogen at ambient temperature and pressure. For this purpose, silica gel, NaY and HY zeolites were saturated with 141.8, 80.8 and 59 weight percent of solvent respectively. Operations with various adsorbents from 20 to 60°C at 600 kPa and from 100 to 600 kPa at 20°C were performed experimentally. Then, a mathematical model for this process including a set of partial differential equations was developed where LDF approximation for mass transfer and Henry's isotherm were made use of. These equations were solved numerically. Obtained experimental breakthrough data were utilized to construct Henry’s constant through fitting sorption models. The best values for sorption (that is, adsorption) constants were empirically determined to be 0.970, 0.985 and 0.995 for silica gel, NaY and HY zeolites; respectively. Ultimately, amount of H$_2$S sorption was calculated enabling description of desorption (that is, purging) step for this system with a set of partial differential equations. Thus, empirical desorption constant values of 0.744, 0.749 and 0.753 for silica gel, NaY and HY zeolites respectively, were determined through solving the related PDE.

Key words: H$_2$S adsorption, pressure swing adsorption (PSA), mathematical model.

INTRODUCTION

Many industrial plants including natural gas processing, petroleum refining, petrochemical plants, Kraft mills, coke ovens and coal gasifiers generate significant quantities of H$_2$S (Capone and Kroschwa, 1997; Berkow, 1992). In recent years, many investigations have been focused on the removal of H$_2$S from industries, such as sweetening of natural gas, purification of ammonia and methanol synthesis gas, waste water treatment and hydrogen purification (Keshavarz et al., 2008). Furthermore, due to environmental concerns, various approaches such as chemical absorption by alkanolamines (Furhacker, et al., 2003), bioscrubber (Potivichayanon et al., 2006), and the oxidation of H$_2$S in iron-chelate process (Iliuta and Larachi, 2003) have been supported over the years. Biofiltration has recently been recognized as an effective technology for odor treatment. The effectiveness of biofilters lies on the activity of the microbial population and the type of enrichment, during the inoculation step (Duan et al., 2007; Shinabe et al., 2000; Kim et al., 2008). In addition, electrochemical methods have also attracted...
interests of many researchers (Gercel et al., 2008) for such purposes. One other technique calls for removal of H$_2$S by adsorption on various porous material such as activated carbon (Daley et al., 1997), modified activated carbon (Bagreev et al., 2005), γ-Al$_2$O$_3$, modified clay (Thanh et al., 2005), modified zeolites (Bae et al., 2003) and ZnO/Al-SBA-15 which might be effective and extends the research on mesoporous material (Wang et al., 2008a, b). Many of these processes however, are of limited efficiency and have high-energy costs. Utilizing large amounts of solvent and catalyst in traditional removal processes increases the cost of treatment.

To such processes, cyclic operation of PSA with four stages of pressurization, adsorption, depressurization and purge might be applied. In addition, since NMP is widely used as a solvent for H$_2$S in the absorption technology due to its favorable physical properties and good selectivity toward H$_2$S, it is selected for the present study. Here, the adsorbent bed was wetted by the NMP employing a special method. The layer of liquid functions as the separation agent and is referred to as the stationary phase while the solid grains are referred to as the support. It is reminded that such layered material approach has never been applied for a PSA process previously. The liquid layer outside a solid grain provides the separation bed packing a better selectivity and renders easier regeneration. In this process simultaneous absorption and adsorption, thus, might be studied. Furthermore, such selective solvent increases amount of sorption hence accelerates the process. By coating the adsorbent with the solvent, component to be adsorbed on the solid, first dissolves into the liquid (wetting the solid) and then moves on the surface of the solid. This is the basic idea for removing H$_2$S from methane using PSA technique for which process silica gel, HY and NaY zeolite saturated with NMP solvent are employed in this research.

Previously PSA process has been modeled by different researchers (Cruscza et al., 2005; Jain et al., 2003; Choong et al., 2002; Chang et al., 2004; Bitzer and Zeitz, 2002). However, these studies were performed in two phase of solid and gas. Mass transfer was modeled in three regions of bulk of gas, film of gas around the solid and porous surface of solid. On the other hand, PSS experimental investigations have been performed in a three phase system including solid, liquid and gas in which the porous media is filled with liquid solvent. Anybody was not up to date; no research group has attempted to mathematically model this process. It is experimentally shown that H$_2$S moved from the gas phase onto the sorbent at an elevated pressure, and moved back from the saturated sorbent to the gas phase at a reduced pressure. However, it is reiterated that adsorbent function is indeed more than an inert carrier. It enlarges not only the interface between gaseous and stationary phases, but also performs as an adsorbent (Zhong et al., 2004). However, successful application of this method on industrial scale depends upon correct selection of the adsorbent, suitable preparation of the wet bed, proper development of sorption cycles and suitable operational conditions. Determining solubility is a very significant step toward designing separation process by the PSA technology. In this direction, a bench scale setup capable of providing in-situ breakthrough data was constructed to obtain experimental sorption values for the complete PSA cycle on several adsorbents under different operating conditions. This was followed by developing a mathematical model to simulate and validate the experimental data performed.

MATERIALS AND METHODS

Materials
The feed gas was prepared through mixing of 99.9 wt% pure methane and gaseous H$_2$S. The hydrogen sulfide, itself, was produced through the following reaction:

$$Na_2S(s) + 2HCl(l) \rightarrow NaCl(s) + H_2S(g).$$ (1)

For which, the Merck hydrochloric acid (37 mole%) and commercial powder of sodium sulfide were contacted. This reaction instantly proceeded to completion. Nitrogen gas of 99.9 wt% purity was also purchased from Roham Gas Company. Adsorbents including Blue Merck silica gel, HY and NaY zeolites were acquired from Zeolyst Company and their specifications are listed in Table 1.

Apparatus
The equipment applied in this research consisted of three parts including the reactor to make H$_2$S, the dosing vessel and the PSA apparatus.

The feed for this process was prepared in the dosing vessel by mixing both H$_2$S and pressurize methane. Concentration of the feed was determined by setting its pressure. It is of significance to mention that the feed should be produced at a constant concentration and in adequate amount due to safety reasons. In other words, the amount of feed should be such that the supply to the process is at constant concentration and pressure. Eventually, while the operation pressure was 600 kPa and the flow rate was set to 150 cm$^3$/min, the feed into the dosing vessel was determined to be 2000 kPa. The schematic PSA apparatus is illustrated in Figure 1.

Operation of the modified PSA system is performed as follows. Sodium sulfide powder entered into the reactor from the valve v5. Reaction between sodium sulfide and hydrochloric acid is instantaneous and produces H$_2$S. Care should be taken concerning the reactor pressure in order to prevent the flow of unreacted HCl. Therefore, a mixture of stoichiometric ratio of HCl and pressurized methane was introduced into the D1 vessel and injected instantaneously into the reactor from v4 valve. H$_2$S is then produced in this reactor, where pressure was monotorized with gauge p2.

The mixture of H$_2$S and high pressure methane had to be prepared in the dosing vessel to feed the PSA process. Thus, methane at 2000 kPa of pressure flows into the dosing vessel through valves v2, v6 and v8. This pressure was measured with gauge p3. The feed flow from the dosing vessel was regulated with R2. The sorption column consisted of a 25 cm long Pyrex glass with 1.3 cm of diameter. The operation pressure was set at 600 kPa which is the maximum strength allowed by the glass column. The
Experimental procedure

Preparation of the wet bed

Two methods were utilized to prepare the wet beds for the PSA process. When solution of NMP and acetone were added to powders of NaY and HY zeolites, these particulate matters coagulated to form hard pulp, making impossible the uniform gas flow through them. As such, the solvent sorption on these materials had to be dealt outside of the adsorbent bed, then crushed and loaded into the column. However, for the silica gel materials no such problem was encountered when adding solutions of NMP and acetone. Preparation of such wet beds was performed as follows:

Preparation of the wet silica gel bed

The amount of the NMP solvent needed was defined as the weight percent of this material in reference to the bed weight. A mixture of 50 wt% NMP and acetone was prepared and added to the bed. The boiling point of the NMP and acetone are 202 and 60°C, respectively. The temperature was set at 80°C, the liquid in the column began to boil and acetone evaporated out. The silica gel particles were coated and wetted by the NMP when the solution was brought to boil. The solution and then the powder were continuously added until the whole bed was perfectly wetted. Finally, the excess liquid was drained out.

The adsorption bed with 141.8 wt% of the NMP solvent was prepared. The Merck silica gel is blue. When exposed to gases, turns to white which is a sign of adsorption operation taking place. Obviously, the more liquid solvent adsorbed on the solid, the higher the solid capability will be to separate the H$_2$S from the gaseous mixture. It is noteworthy that, utilization of such porous adsorbent has the advantage of providing much greater adsorption surface area than any other gas absorption process (Keshavarz et al., 2008).

Preparation of the wet zeolite bed

NaY and HY zeolite samples were separately immersed into a solution of NMP (25 wt%) in acetone. The glass beaker containing the sample was placed in a water bath at 80°C, allowing a slow evaporation of the acetone. The addition of both NMP and acetone was repeated until excess liquid remained in the beaker. This means that the resulting adsorbent was saturated with the NMP.

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**Table 1. Structural specification of the adsorbents applied in this study.**

<table>
<thead>
<tr>
<th>Specification</th>
<th>Silica gel</th>
<th>NaY zeolite</th>
<th>HY zeolite</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bulk density (g/cm$^3$)</td>
<td>0.98</td>
<td>0.215</td>
<td>0.289</td>
</tr>
<tr>
<td>BET surface area (m$^2$/g)</td>
<td>900</td>
<td>730</td>
<td>550</td>
</tr>
<tr>
<td>Average pore size (nm)</td>
<td>32</td>
<td>24.65</td>
<td>24.5</td>
</tr>
<tr>
<td>Pore volume (cm$^3$/g)</td>
<td>0.7</td>
<td>0.34</td>
<td>0.26</td>
</tr>
<tr>
<td>Si/Al ratio</td>
<td>-</td>
<td>5.1</td>
<td>5.1</td>
</tr>
</tbody>
</table>

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**Figure 1. PSA apparatus built for the study.**
Table 2. Weight percent fixed of the zeolite adsorbents with NMP.

<table>
<thead>
<tr>
<th>Adsorbent</th>
<th>NMP wt%</th>
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<tbody>
<tr>
<td>Zeolite NAY</td>
<td>80.8</td>
</tr>
<tr>
<td>Zeolite HY</td>
<td>59.0</td>
</tr>
</tbody>
</table>

Figure 2. Breakthrough behavior of H₂S on various adsorbents at p = 600kPa and T = 298 K.

and its grains were coated by the solvent. The quantity of NMP coated on the solid surface was determined from sample weights before and after this procedure. The NMP concentration on the adsorbent was defined as the weight percentage of the NMP per unit weight of zeolite. The amount of solvent needed to fixed on the adsorbents is illustrated in Table 2.

Wet column operation

The desired sorption is the one taking place through the bed and having not only a good separation efficiency, but also an easy adsorbent regeneration by using a gas, such as nitrogen, as purge. Thus, experiments with different adsorbents were performed under various operating pressures and temperatures. To begin with, breakthrough curves with different adsorbents were determined. These are illustrated in Figure 2. It is seen that, due to getting more easily wetted than zeolites, silica gel is more efficient in retaining H₂S than NaY and HY zeolites.

Furthermore, this figure illustrates that NaY is more efficient than HY zeolite which is in agreement with the work of other researchers as well (Cosoli et al., 2008a, b). This result is due to the fact that the NaY has more surface area and wider pore size distributions than HY zeolite (Table 1). These data indicate that suitable wetting with the NMP solvent and the preparation technique for the wet bed affects the bed efficiency, and hence the amount of hydrogen sulfide adsorbed on the process. When exhaust concentration reaches feed concentration, it means that the bed is saturated. So amount of sorption with known flow rate and concentration of feed and integrated surface above breakthrough curve with equation (2) were calculated.

\[
q = \frac{QC_0}{m} \int_0^t \left(1 - \frac{C}{C_0}\right)dt
\]  

(2)

Figure 3. Hydrogen sulfide adsorbed at various temperatures and at 600 kPa with flow rate 150 cm²/min and C₀ of $2 \times 10^{-5}$ (g/cm³) on a) silica gel, b) NaY zeolite and c) HY zeolite.

Effect of temperature

Breakthrough curves for each adsorbent at different temperatures and at 600 kPa of pressure are presented in Figure 3. It is generally concluded that with an increase in temperature the amount of
adsorption decreases. This temperature effect is rationalized due to the solvent amount coated on a particular adsorbent. This amount increased from the HY to NaY zeolite, and further to the silica gel material. Amount of sorption calculated with equation (2) to different temperature for each adsorbent is illustrated in Table 3.

**Effect of pressure**

Breakthrough curves for each adsorbent at different pressures and under a constant temperature are illustrated in Figure 4. It is observed that pressure imposes significant effects on the adsorption property of these materials. This parameter has a more pronounced effect on the adsorption of the silica gel than on NaY and HY zeolites, respectively. Once again, due to more amount of the NMP solvent adsorbed onto the silica gel, this adsorbent has higher capability to separate higher amounts of H\textsubscript{2}S compared to NaY and HY zeolites. Amount of sorption calculated with equation (2) to different pressure for each adsorbent is illustrated in Table 4.

**THEORY**

The proposed theoretical model considers a separation system for sorption of H\textsubscript{2}S through the PSA process in a packed column (that is, a fixed bed). The following main assumptions are made: i) ideal gas behavior, ii) axially dispersed plug flow, iii) uniform cross-sectional void fraction, iv) uniform adsorbent properties along the axial coordinate, v) negligible pressure drop and vi) negligible radial gradients which are all in accordance with previous works (Cruz et al., 2005; Cruz et al., 2003). With the gaseous mixture flowing over the packed bed, H\textsubscript{2}S dissolves into the liquid layer surrounding the packing material. According to the aforementioned assumptions, the mathematical model equations for an element of the bed with length dz may be written as follows. The component material balance is given by:

$$ \frac{\partial c_i}{\partial t} + u \frac{\partial c_i}{\partial z} - D \frac{\partial^2 c_i}{\partial z^2} + \left(1 - \frac{\varepsilon}{\varepsilon_i}\right) \frac{\partial q_i}{\partial t} = 0 $$

(3)

In which, \(c_i\) is the \(i\)th component molar concentration in the fluid phase, \(\varepsilon\) is the bed void fraction (ratio between the free volume and the total volume), \(u\) is the interstitial molar velocity, \(z\) is the spatial coordinate, \(D\) is the effective axial dispersion coefficient, \(t\) is the time variable and \(q\) is the mass adsorbed per unit mass of the sorbent particles. It is supposed that the liquid solvent coated the whole particle including the pores and outside surface (that is; due to the saturation wt\% discussed earlier). Thus, the amount of hydrogen sulfide adsorbed to the surface of this particle will be equal to the amount adsorbed to the liquid layer surrounding the particle and the effective axial dispersion coefficient for diffusion of gas into liquid [wilke-chang] (Suzuki, 1990) was formulated as follows:

$$ D_e = 7.4 \times 10^{-8} \left(\alpha M\right)^{\frac{1}{2}} T / \left(\mu l V_1^{0.6}\right) = 6.34 \times 10^{-4} $$

(4)

**Table 3.** Amount of adsorption to different temperature for each adsorbent in 600 kPa.

<table>
<thead>
<tr>
<th>Temp (K)</th>
<th>Silica gel</th>
<th>NaY</th>
<th>HY</th>
</tr>
</thead>
<tbody>
<tr>
<td>293</td>
<td>5.4 × 10^{-4}</td>
<td>4.44 × 10^{-4}</td>
<td>3.2 × 10^{-4}</td>
</tr>
<tr>
<td>313</td>
<td>5.36 × 10^{-4}</td>
<td>4.2 × 10^{-4}</td>
<td>3.12 × 10^{-4}</td>
</tr>
<tr>
<td>333</td>
<td>5.28 × 10^{-4}</td>
<td>3.7 × 10^{-4}</td>
<td>3.04 × 10^{-4}</td>
</tr>
</tbody>
</table>

**Figure 4.** Hydrogen sulfide adsorbed at various pressures and at 293 K. Feed flowrate was 0.003, 0.0024 and 0.002 (gr/ min) at 600,300 and 100 (kPa) respectively on a) silica gel, b) NaY zeolite and c) HY zeolite.
Table 4. Amount of adsorption to different pressure for each adsorbent at 293 K.

<table>
<thead>
<tr>
<th>Pressure (kPa)</th>
<th>Silica gel</th>
<th>Nay</th>
<th>Hy</th>
</tr>
</thead>
<tbody>
<tr>
<td>100</td>
<td>$4 \times 10^{-4}$</td>
<td>$3.12 \times 10^{-4}$</td>
<td>$2.2 \times 10^{-4}$</td>
</tr>
<tr>
<td>300</td>
<td>$4.48 \times 10^{-4}$</td>
<td>$3.68 \times 10^{-4}$</td>
<td>$2.6 \times 10^{-4}$</td>
</tr>
<tr>
<td>600</td>
<td>$5.4 \times 10^{-4}$</td>
<td>$4.44 \times 10^{-4}$</td>
<td>$3.2 \times 10^{-4}$</td>
</tr>
</tbody>
</table>

Here “a” represents the association coefficient taken to be 1 for non-associative solvents, $M$ and $\mu$ are molecular weight and viscosity of the solvent; respectively and $V_1$ is the molar volume of the solvent. In addition, the last term of the equation (2) was determined as follows:

$$\frac{\partial q_i}{\partial t} = k_j (c - c^*)$$  \hspace{1cm} (5)

Here, $k_j$ is the mass transfer coefficient determined from equation (6) (Wakao and Funazkri, 1978), that is;

$$N_{2a} = k_j d_p \frac{d_x}{D} = 2 + 1.1 \left( \frac{\mu}{\rho D} \right) \left( \frac{d_x G}{\mu} \right)^{0.6} = 2 + 1.1 (Sc)^{1/3} (Re)^{0.6}$$  \hspace{1cm} (6)

In which, $\rho, \mu$ were taken to be $7.6 \times 10^{-3}$ and, $1.63 \times 10^{-4}$ respectively. Furthermore, $d_p$ for silica gel was $9 \times 10^{-4}$ cm, for NaY zeolite was $1 \times 10^{-4}$ cm and for HY zeolite was taken to be $5 \times 10^{-5}$ for $hy$; with the initial condition of:

$$c(t, z) = 0 \text{ for } t = 0,$$  \hspace{1cm} (7)

and boundary conditions of:

$$c(t, z) = c_f \text{ for } z = 0$$  \hspace{1cm} (8)

$$\frac{\partial c(t, L)}{\partial z} = 0 \text{ for } z = L$$  \hspace{1cm} (9)

And $c^*$ described with the Henry’s law as follows:

$$c_i^* = Kc_i$$  \hspace{1cm} (10)

MATHEMATICAL MODEL RESULTS AND DISCUSSIONS

Adsorption constants

The PSA process under considerations was simulated for each adsorbent. The accuracy of the model results was estimated through $R$ the following equation:

$$R = \frac{1}{N} \sqrt{\sum (y_{exp} - y_{mod})^2}$$  \hspace{1cm} (11)

Here, $y$ is the exhaust concentration and $N$ is the number of adsorption points studied. To determine the best adsorption behavior, first experimental data is obtained at 293 K and 600 KPa for hydrogen sulfide adsorption on various adsorbents. $K$ is 0.97, 0.985 and 0.995 for silica gel, NaY and HY zeolites; respectively. Experimental and model data are shown in Figures 5a – c.

Determination of the breakthrough capacity

In order to simulate the complete cyclic PSA process, amount of $H_2S$ sorbent in the adsorption step should be removed from the bed through the purge step. This is calculated from equation (2). Ultimately, the respective mass of $H_2S$ adsorbed was calculated as follows:

$$m_{H_2S} = q^* m_s$$  \hspace{1cm} (12)

Simulation of the purge step

Experimentally, a stream of pure nitrogen was employed counter currently to the feed step, in order to remove the $H_2S$ from the adsorbent into a safe container. In the simulation work, the following assumptions were considered. The diffusion term was neglected and, for any element of length $dz$, two inputs were included: one from bulk flow of gas and the other to be the amount of $H_2S$ entering into each element $dz$. The mass balance equation is, therefore, as follows:

$$\frac{\partial c_i}{\partial t} = u \frac{\partial c_i}{\partial z} + \left( \frac{1 - \varepsilon}{\varepsilon} \right) k_c_i$$  \hspace{1cm} (13)

For which the boundary conditions were:
Figure 5. Experimental and theoretical data for hydrogen sulfide adsorption on various adsorbents: a) on silica gel \((R = 0.348, K = 0.97)\), b) on NaY zeolite \((R = 0.41, K = 0.985)\) and c) on HY zeolite \((R = 0.49, K = 0.995)\).

Figure 6. Comparison of simulation and experimental data for the purge step over the different adsorbents at atmospheric pressures and 293 K.

\[
c_i(0) = 0 \quad \text{for} \quad z = 0 \\
\frac{\partial c_i}{\partial z} = 0 \quad \text{for} \quad z = L
\]

and the initial condition was set such that the bed was divided into \(m\) parts in each of which the mass element was taken to be as follows:

\[
\Delta m = \frac{m_{H_2S}}{m}
\]

The \(H_2S\) concentration in each element is thus given by,

\[
C_{(H_2S)} = \frac{\Delta m}{\varepsilon(A\Delta z)}
\]

Which is the hydrogen sulfide concentration in each element at \(t = 0\). This partial differential equation was solved utilizing the method of line (that is, MOL) routine. Comparisons of the simulated response with the experimental data are illustrated in Figure 6.

It is seen through Figure 6 that once again the best comparison between experimental and theoretical data is obtained in the following order: silica gel, NaY zeolite and HY zeolite. This is due to the amount of NMP solvent adsorbed on each of these materials discussed previously. Ultimately, the desorption constants of all these materials through solving equation (11) numerically are determined to be equal to 0.744, 0.749 and 0.753 for silica gel, NaY and HY zeolites, respectively. These results
emphasized the role of solvent selected for the PSA process.

**Conclusion**

Utilizing the PSA technique to separate a gaseous mixture to its components is considered to be a new route with low costs of primary construction and operation compared with other methods of separation. The idea of coating the dry adsorbent with a selective solvent opens a new prospect in PSA process utilization. Besides choosing an efficient adsorbent, one may make use of an efficient solvent to make the process more flexible. Utilization of the solvent assisted technology in boiling and wetting of the adsorbent may create a suitable yield and efficiency for the bed. Furthermore, use of solvent made rapid adsorption and desorption of the bed to be advantageous.

In this study, an experimental setup was constructed in order to be capable of in situ breakthrough measurements of hydrogen sulfide and methane gas mixtures in a packed bed. This was followed by developing a mathematical model to describe the behavior of this system. In this direction, several adsorbents, including silica gel, NaY and HY zeolites, were employed for which sorption and desorption constants were calculated through proposing a rather general isotherm. The present research has paved down the road to analyze the separation behavior of systems affected by pressure swing which may be employed to optimize such processes.

**ACKNOWLEDGMENT**

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**REFERENCES**


<table>
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<tr>
<th><strong>Nomenclature</strong></th>
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<td>b bed</td>
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<tr>
<td>c</td>
<td>f feed</td>
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<td>c&lt;sub&gt;r&lt;/sub&gt;</td>
<td>g gas</td>
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<td>D&lt;sub&gt;ax&lt;/sub&gt;</td>
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