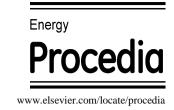


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New process concepts for CO₂ capture based on precipitating amino acids.

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Abstract

This work summarises the results of the design of novel separation processes for CO_2 removal from flue gas based on precipitating amino acid solvents. The processes here described (DECAB, DECAB Plus and pH-swing) use a combination of enhanced CO_2 absorption (based on the Le Chatelier's principle) and / or enhanced CO_2 desorption (based on pH-shift), which contribute substantially to the reduction of regeneration energy. An evaluation of these processes has been developed based on literature data, thermodynamic principles and experimental data. As solvent example, the potassium salt of taurine was selected. The results show that these processes are environmentally friendly (no emissions to the air, lower ecotoxicity) and have lower energy consumption than conventional MEA. The DECAB Plus process has been identified as the option with lower energy consumption (66% of conventional MEA- ie ca 2.4 GJ/t CO_2). This study has established the potential of precipitating amino acids as an energy effective alternative to alkanol amines. The future work will focus on determining the cost of CO_2 avoidance and investigating other solvents that will further lead to benefits over conventional processes.

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Keywords: Amino acid salts, post combustion capture, process evaluation

Nomenclature

MEA monoethanolamine KTau potassium taurate PCC post-combustion capture

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1. Introduction

Amino acid salts have been used for acid gas removal since the 30s. Several amino acid salt based processes have also been developed for post combustion capture (PCC) of carbon dioxide from flue gas. One example of these is the process provided by Siemens, which has energy consumption around 73% of the conventional MEA process [1]. TNO has also investigated processes based on the potassium salt of certain amino acids, showing increased stability and resistance to degradation over conventional MEA [2]. They are attractive solvents for PCC due to their unique properties over alkanol amines such as: low environmental impact and high biodegradability [3], negligible volatility and high resistance to oxidative degradation [2]. Therefore, interest has grown in the performance of different amino acid salts for PCC application and the CO₂ absorption characteristics for common amino acids are under extensive investigation. Several researchers have reported the CO₂ absorption on sarcosine, glycine, alanine, taurine and proline solutions [4-7]. Also properties that are necessary for mass transfer evaluation, such us density, viscosity, CO₂ diffusivity, N₂O solubility and solvent kinetics have been investigated and reported for the mentioned amino acids under different conditions [8-13].

Amino acids react with CO_2 like alkanol amines, having the same reaction mechanism, but there are differences between them in PCC application. Fig. 1 illustrates a conventional amine based capture process. This process generally consists of an absorption column, where the CO_2 is chemically bond to the alkanol amine. Depending on the type of amine different species are predominantly formed. In the case of primary amines carbamate is predominantly formed according to the equilibrium reaction (1):

$$CO_2 + 2R - NH_2 \leftrightarrow R - NHCOO^- + R - NH_3^+$$
 (1)

The CO_2 loaded product is extracted at the bottom of the absorption column. The remaining gas is clean and can be vented to the atmosphere after a final wash in order to avoid solvent evaporation. The loaded solvent is further processed in a stripper, where it can be thermally regenerated for further use:

$$R - NHCOO^{-} + R - NH_{3}^{+} + \longleftrightarrow_{Heat} CO_{2} + 2R - NH_{2}$$
 (2)

However, unlike amines, amino acids form electrolytes in aqueous solution that comprise a zwitterion. In order to react with CO_2 , this zwitterion species needs to be neutralised with a strong base. The reactions between CO_2 and amino acid salts are illustrated in fig. 1, assuming potassium hydroxide as strong base. Due to the limited zwitterion solubility in water, which is a function of pH, and the strong electrolyte characteristics of the amino acid salt solutions, precipitates may be formed when solution's pH decreases, as a consequence of CO_2 absorption. For the conventional amine scrubbing process depicted in fig. 1, which is not designed to handle solids, this physical behaviour limits the concentration of active amino acid in the solvent.

Nevertheless, there are characteristics specific to amino acid salts that contribute to decrease the energy consumption of the capture process influencing the equilibrium reaction ((1) and (2)) by other means than heat. TNO has investigated different process alternatives which include precipitation in the process [14]. Different precipitate types can be encountered depending on the amino acid structure and solubility. Amino acids with a primary amino group, such as taurine, precipitate only the zwitterion species [15]. In other cases, the precipitate formed is potassium bicarbonate. This is the case of amino acids with a hindered amino group and relatively high zwitterion solubility, such as proline [16]. Independently of the precipitate type, the formation of a solid reaction product during absorption and its removal from the solution phase by precipitation shifts the reaction equilibrium towards the production of more products (carbamate or bicarbonate). This effect has been indicated in fig. 1 (enhanced absorption),

where the possible precipitating species are highlighted. Moreover, the formation of the precipitate allows for phase separation (solid from liquid) and formation of a new rich solution, in which acidity is enhanced by manipulating the electrolytes in the solution. For instance, in the case that the amino acid is concentrated in the solid phase, the effect of partial removal of the solution will result in a higher reduction of K^+ compared to the reduction in amino acid. After dissolution of the solid phase, this will induce a lower pH in the rich solution that is treated in the stripper due to a higher ratio of amino acid to K^+ . The lower pH promotes the hydrolysis of the carbamate species, resulting in the formation of amino acid salt and bicarbonate, which revert to the zwitterion species and carbon dioxide at lower pH. This effect has also been indicated in fig. 1 (enhanced desorption). As net effect, a lower pH during desorption will shift the CO_2 reaction equilibrium towards the release of CO_2 .

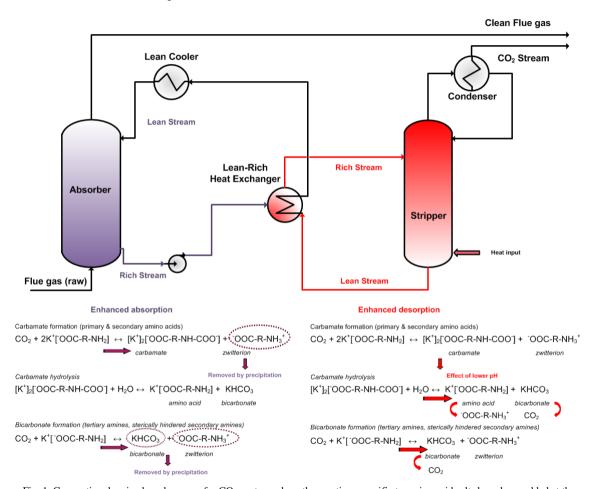


Fig. 1. Conventional amine based process for CO_2 capture where the reactions specific to amino acid salts have been added at the bottom of the absorber and the stripper. Besides the heat input necessary to regenerate the solvent, in the case of precipitating amino acids two more effects are possible: Enhanced absorption (purple) due to the precipitation of reaction products during absorption and enhanced desorption (red) due to a lower pH that results from increasing the amino acid to of K^+ ratio in solution.

Naturally, there are counter acting factors in these phenomena. The presence of solids in the process requires the selection of different contactor equipment for absorption, such as a spray tower, for which mass transfer characteristics need to be addressed. Also, the strong electrolyte nature of the amino acid salt solutions might have an effect in absorption kinetics that needs to be considered for optimum design. Moreover, in order to induce a pH shift during desorption, a new unit operation needs to be included, which separates the precipitate and part of the supernatant. The latter is recycled to the absorption column with an effect on solvent capacity since the supernatant also contains carbonated species.

This work provides the evaluation of different process concepts based on the combination of enhanced absorption and desorption, considering all phenomena and factors involved. The selected solvent for the evaluation of these concepts is 4M potassium taurate. The strategy followed is based on the investigation of the key properties and parameters that govern the absorption and desorption in precipitating amino acids. The effects of precipitation during absorption and pH-shift during desorption were verified experimentally. The experimentally derived properties were used for conceptual design and process modelling, based on semi-empirical formulas. Three different process concepts are provided in this work: DECAB, DECAB Plus and pH-Swing. Finally, the evaluation of these processes gives the trade–off between all phenomena and shows the differences between the selected processes and conventional MEA.

2. Experimental verification of enhanced absorption and enhanced desorption

2.1. Effect of amino acid precipitation during absorption

The effect of amino acid precipitation of CO_2 absorption has been investigated for potassium taurate solutions. Fig. 2 shows the absorption isotherms for potassium taurate solutions of different concentrations. The precipitation effect can be seen in the experimental data of Kumar at 25°C ([5], [15]), when comparing the specific loading of potassium taurate solutions at constant CO_2 partial pressure and different concentrations of amino acid salt. For instance, based on a constant CO_2 partial pressure of 1 kPa, the specific loading at 2M, with no precipitates present, is lower than the specific loading at 3M, with precipitates present. However, in the absence of precipitation the expected result would have been the opposite (i.e. higher specific loading for the 2M solution versus the 3M solution) due to the salting out effect induced by the higher ionic strength of more concentrated solutions. The same trend can be observed at 40°C (this work), where the specific loading of the 3M solution with no precipitates (ca. 1 kPa) is lower than that of 4M solution with precipitates present, but becomes higher when precipitates are formed (especially visible ca 10 kPa).

2.2. Effect of lower pH during desorption

The effect of pH was investigated by CO_2 absorption measurements on asymmetric solutions of taurine and KOH (i.e solutions containing different molar amounts of amino acid and base). Results in fig. 3 represent the absorption isotherms for different asymmetric solutions of potassium taurate and different temperatures. These results show that changes in the molar ratio Taurine/KOH can have a substantial influence in the partial pressure of CO_2 in equilibrium with the solution. One general trend observed at every temperature is that the loading decreases with decreasing KOH concentration (i.e. lower pH), at constant partial pressure of CO_2 . In other words, for a given loading, higher partial pressures of CO_2 will be obtained during regeneration when the molar ratio Taurine to KOH increases. This result verifies the pH effect illustrated in fig. 1 (i.e. equilibrium is shift towards the release of CO_2).

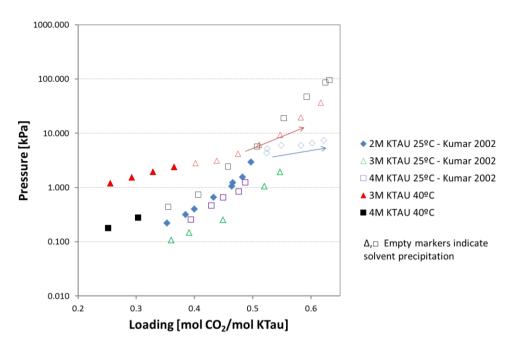


Fig. 2. Absorption isotherms of CO₂ over different solutions of potassium taurate. The concentration in the legend indicates the total concentration of amino acid and potassium in solution and the absorption temperature. The presence of solids in solution is indicated with empty markers. The arrows in the figure help to show the effect of solvent precipitation

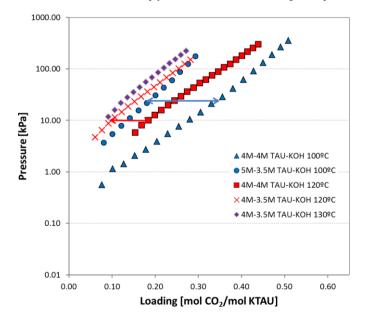


Fig. 3. Absorption isotherms of CO₂ over different solutions of potassium taurate. The legend indicates the total concentration of amino acid (first) and potassium hydroxide (second) in solution and the absorption temperature. The arrows in the figure help to show the effect of pH.

3. DECAB process concept

Fig. 4 shows the schematic representation of the DECAB process concept [14]. The flue gas (at 40°C) is contacted with pre-loaded solvent in a spray-tower. The spray-tower consists of a column with no internals. The solvent is sprayed as fine droplets with a high surface area for contacting the gas with the solvent. As a result of this contact, the CO₂ undergoes a chemical reaction with the solvent that leads to the formation of carbamate and carbonate ions (as shown in fig.1). As a consequence of this reaction, the pH of the solvent solution decreases and so does the solubility of the amino acid. When the solubility limit is reached, the amino-acid precipitates as an amino-acid zwitterion. The resulting slurry is collected at the bottom of the tower with a loading of 0.48 mol CO₂/mol KTau. The partial pressure of CO₂ in the flue gas is decreased until the critical point for precipitation of the amino acid (0.35 mol CO₂/mol Ktau). The remaining CO₂ in the gas is captured in the absorption column, where the depleted flue gas is contacted with lean solvent. The absorption column is a conventional packed absorption column filled in with structured packing. There, the CO₂ partial pressure is reduced to the desired value for 90% CO₂ removal. The rich stream containing the taurine solids, is further processed in the stripper, via the lean-rich heat exchanger, to release the CO₂. The lean-rich heat exchanger also needs to be of a suitable type to handle solids (e.g. spiral heat exchanger).

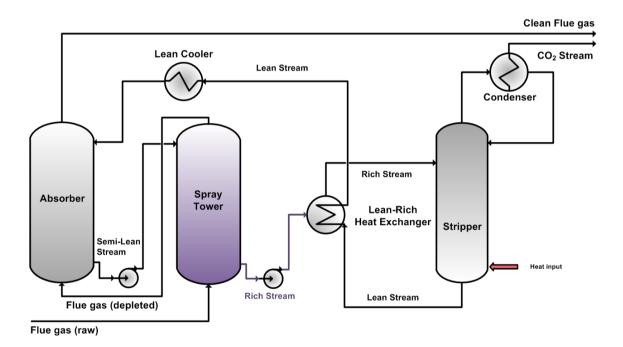


Fig. 4. DECAB process concept for CO₂ capture. Enhanced absorption due to the precipitation of reaction products during absorption is highlighted in purple.

4. DECAB Plus process concept

This enhanced desorption concept can be achieved by the process schematically presented in fig. 5. In this case, the flue gas is treated with a solution of potassium taurate containing 1:1 molar amounts of Taurine and KOH in a spray tower contactor or a sequence of spray tower contactor and packed column. The result is a rich stream in the form of slurry that contains taurine solids and a supernatant with the carbonated and potassium species. This slurry is treated in a solid-liquid separator (hydrocyclone or decanter) to partially separate the supernatant from the solids. The resulting slurry, enriched in taurine, is further processed in the stripper for desorption. The supernatant excess, enriched in potassium ions, is recycled to the absorber without passing through the stripper. In this process, the pH of the rich solution is decreased before desorption (after dissolving the taurine crystals), leading to easier release of the chemically bonded CO₂. However, in order to induce a pH shift during desorption, solvent capacity may be negatively affected since part of the remaining supernatant (containing carbonated species) is recycled to the absorption process. It is of importance to highlight that dissolving the taurine crystals in both DECAB and DECAB Plus process concepts requires the input of low grade energy. This energy has a different quality to the steam required in the reboiler (hot water of around 80°C could be used for this purpose). This could be easily integrated into the power plant by using the reboiler's condensate prior to its integration into the preheaters train. For this reason, this energy has not been included in the energy analysis presented in this work.

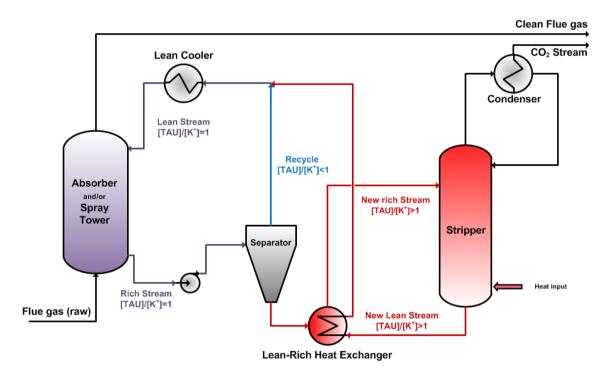


Fig. 5. DECAB Plus process concept for CO_2 capture. Enhanced absorption due to the precipitation of reaction products is highlighted in purple and enhanced desorption due to the induced lower pH for regeneration is highlighted in red. The ratio of the total taurine concentration to the total potassium concentration in solution is indicated below the name of each stream. This ratio can be 1 (equimolar solution), higher than 1 (solution with less potassium than the equimolar and , therefore lower pH) or less than 1 (solution with more potassium than the equimolar solution and, therefore, higher pH)

5. pH-swing process concept

The process is schematically presented in fig. 6. In this case, the flue gas is treated with a solution of potassium taurate containing 1:1 molar amounts of Taurine and KOH in a conventional packed column. Absorption takes place at a temperature around 60°C in order to avoid taurine precipitation. The resulting stream is cooled to temperatures below 30°C to form the slurry that contains taurine solids and a supernatant with the carbonated and potassium species. The slurry is then treated in a solid-liquid separator to partially separate the supernatant from the solids. The resulting slurry, is more enriched in taurine than in the DECAB Plus case, and is further processed in the stripper for desorption. The supernatant excess, enriched in potassium ions, is recycled to the absorber. The main difference between this process concept and the DECAB Plus is that absorption occurs at higher temperatures (reducing solvent capacity) but at the same time the pH of the rich solution is further decreased, leading to a more regenerated solvent, and less CO₂ is recycled to the absorber. The last two factors contribute to recover the capacity loss due to the absorption at higher temperatures. Moreover, this process concept does not require the use of a spray tower.

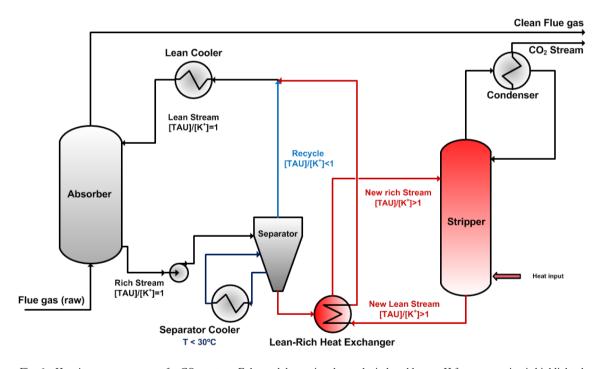


Fig. 6. pH-swing process concept for CO_2 capture. Enhanced desorption due to the induced lower pH for regeneration is highlighted in red. The ratio of the total taurine concentration to the total potassium concentration in solution is indicated below the name of each stream. This ratio can be 1 (equimolar solution), higher than 1 (solution with less potassium than the equimolar and , therefore lower pH) or less than 1 (solution with more potassium than the equimolar solution and, therefore, higher pH)

6. Key facts from process evaluation

This section summarises the evaluation results of the process concepts presented in sections 3 to 5. The strategy followed in this evaluation is based on the compilation and determination of the key properties and parameters that govern the absorption and regeneration of the solvent. The following properties have been investigated experimentally:

- Amino acid solubility as a function of temperature and pH.
- Slurry weight (fraction of the rich stream that contains solids) as a function of loading and temperature.
- Vapour pressure at different KTau concentrations.
- Vapour Liquid Equilibria at different temperatures and different TAU/K⁺ ratios.
- Absorption fluxes at different temperatures and concentrations.

The assessment of the technical performance has been carried out by a process model implemented in a spreadsheet application. The model is based on equilibrium; therefore the absorption fluxes have been compared separately but are not used in the estimation of mass and energy balances. The key facts for each process concept, based on 4M KTau, are presented in fig. 7 and compared to the benchmark. As benchmark, a conventional 5M MEA process was also evaluated with the developed equilibrium model, and based on the same amount of experimental data as in the case of KTau. The MEA process model was verified with experimental data from pilot plant experiments [17].

All the process concepts evaluated show less specific energy consumption than the MEA benchmark [3.7 GJ/t CO₂]. This reduction is mainly due to the lower absorption heat of the processes and the lower $P_{-H2O/CO2}$ ratio (ratio of water vapour pressure to CO₂ partial pressure at the top of the stripper). The absorption heat represents the averaged absorption heat from lean to rich conditions and it was estimated with the process model for all the process concepts. The results show that the process concepts that induce a higher pH-shift (i.e. pH-swing) also have much lower absorption heat. Compared to MEA, the absorption heat is about 40% of the MEA benchmark. The $P_{-H2O/CO2}$ ratio was also estimated with the process model for all the concepts evaluated and is also lower than that of MEA, due to the electrolyte concentration of the potassium taurate solutions. Despite the fact that the solvent capacity of the process concepts evaluated is lower than the MEA benchmark the other factors (absorption heat and $P_{-H2O/CO2}$ ratio) compensate the capacity loss, contributing to reduce the energy of regeneration.

One of the issues of the process concepts evaluated is the low absorption fluxes. These were experimentally derived from absorption experiments in a double stirred cell. The fluxes are estimated at lean loading and 40°C conditions for MEA, DECAB and DECAB Plus processes, and at 60°C for the pH-swing process. The results in fig.7 show absorption fluxes about half of MEA for the DECAB and DECAB Plus concepts. This is partially overcome in the pH-swing concept by absorbing at higher temperatures (63% of MEA absorption flux). Nevertheless, the DECAB and DECAB Plus processes use a spray tower for absorption which generally creates more interface area for mass transfer than packed columns [18] and could partially compensate the lower flux. Therefore, a detail mass transfer study and economic evaluation need to be performed in order to establish the economic benefit of these process concepts, where a substantial reduction of regeneration energy is achieved, but at the expense of lower absorption fluxes that may increase the capital expenditure.

There are also two important factors that are included in the evaluation (eco-toxicity and corrosion) that add value to the process concepts evaluated. Eco-toxicity estimation is based on the data from Eige-Haugmo [19] and compares the EC-50 (mg/l) for MEA and the average of three different amino acids (Glycine, Sarcosine and alanine). Generally, the eco-toxicity of amino acids is substantially lower than MEA, although the specific values for taurine are not reported.

Corrosion estimations are based on the data from Ahn and co-authors [20] and compare the corrosion rate of KTau to MEA (at the same concentration and temperature) for carbon steel coupons. The corrosion of potassium taurate solutions is about 40% of that of MEA. This indicates that although the design of the KTau process concepts will include larger equipment (especially for the absorption tower) the associated materials may be cheaper (carbon steel versus stainless steel) and the magnitude of the maintenance would be substantially lower.

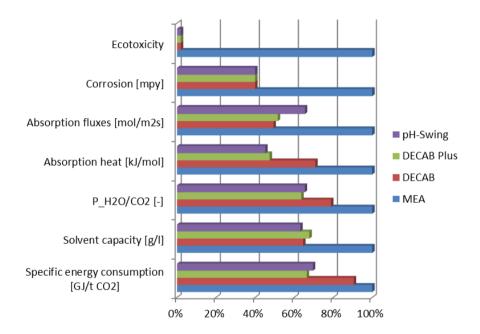


Fig. 7. Comparison between the investigated process concepts, based on 4M potassium taurate, and conventional MEA.

7. Conclusions and future work

This work has evaluated different process concepts that use precipitating amino acid solutions for CO_2 capture. The formation of a solid phase during absorption has benefits for both absorption and desorption. During absorption, the formation of a solid reaction product shifts the equilibrium towards the production of products (carbamate and amino acid zwitterion) resulting in absorption enhancement. The solid – liquid partition can also be manipulated to form a rich stream of lower pH that is processed in the stripper, resulting in enhanced desorption. Both effects, enhanced absorption and enhanced desorption have been

verified experimentally for potassium taurate. Based on the combination of these phenomena, three different process configurations have been investigated (DECAB, DECAB Plus and pH-Swing) based on the potassium salt of taurine. The key advantages of the processes investigated are:

- Low energy consumption: The process concepts investigated have lower reboiler duty than conventional MEA. The DECAB Plus process option has the lowest energy consumption (2.4 GJ/t CO₂) due to a lower vapor pressure of the solvent and a substantial reduction in the absorption heat related to the lower pH during regeneration.
- Low environmental impact: Since the processes investigated are based on the salt of amino acids, there are fewer emissions to the air. Also, the potassium taurate solvent has higher resistance to degradation, resulting in less degradation products for disposal. Finally, the toxicity of amino acids is substantially lower than that of conventional amines.
- Low corrosion: the investigated solvent has been reported to have less corrosive effects on carbon steel than MEA.

The potential to significantly reduce energy consumption by precipitating amino acid solvents has been established. The future work will investigate the mass transfer characteristics and design in the DECAB and DECAB Plus concepts and will explore different options to reduce energy consumption to values just below $2GJ/t\ CO_2$:

- Different process options for regeneration are under investigation. For instance, the lean vapour recompression option and desorption at higher pressures and temperatures are considered.
- Solvent screening procedures have been implemented to improve the base line performance of DECAB. The strategy followed is the selection of appropriate amino acid solvents with higher net loading than taurine (e.g. more hindered amino acids) but similar or higher absorption fluxes.

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