

MULTI-CRITERIA ANALYSIS OF SYNTHESIS METHODS FOR Ni-BASED CATALYSTS

VEČKRITERIJSKA ANALIZA SINTEZNIH METOD NA OSNOVI Ni KATALIZATORJA

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Catalysts based on the Ni/Al₂O₃ system are used in a variety of catalytic processes. Catalysts are commonly synthesized through thermochemical routes (impregnation, precipitation, coprecipitation and others). The authors prepared a Ni-Pd/Al₂O₃ catalyst supported by a ceramic foam, using a novel method, whereby the foam was impregnated with aerosol. This paper evaluates the synthesis methods for the experimentally obtained Ni-Pd/Al₂O₃ catalyst in comparison with other Ni-based catalysts, using three multi-criteria analysis methods (SAW, TOPSIS and PROMETHEE II). The synthesis methods for Ni-based catalysts were compared with respect to the following parameters: preparation method, addition of the precipitation agent during preparation, forming and mixing precursors, filtration, drying procedure, calcination, reduction, and the presence of NiAl₂O₄. The final results indicate that the synthesis method for the Ni-Pd/Al₂O₃ catalyst is the best ranked in comparison with the others.

Keywords: catalysts, multi-criteria analysis, Ni-Pd/Al₂O₃, ceramic foam, aerosol method

Katalizatorji na osnovi sistema Ni/Al₂O₃ se uporabljajo v številnih katalitičnih procesih. Katalizatorji so običajno sintetizirani po termokemijski poti (impregnacija, izločanje, koprecipitacija in drugi). Avtorji so pripravili Ni-Pd/Al₂O₃ katalizator, podprt s keramično peno z uporabo nove metode, kjer je bila pena impregnirana z aerosolom. Članek ocenjuje sintezne metode eksperimentalno dobljenega Ni-Pd/Al₂O₃ katalizatorja z drugimi katalizatorji na osnovi Ni, z uporabo treh večkriterijskih analiznih metod (SAW, TOPSIS, and PROMETHEE II). Sintezne metode katalizatorjev na osnovi Ni, so bile primerjane po naslednjih parametrih: metoda priprave, dodatek izločevalnega sredstva med pripravo, oblikovanje in mešanje prekurzorjev, filtracija, postopek sušenja, kalcinacija, redukcija in prisotnost NiAl₂O₄. Končni rezultati kažejo, da je, v primerjavi z drugimi, najvišje uvrščena metoda sinteze katalizatorja Ni-Pd/Al₂O₃.

Ključne besede: katalizator, večkriterijska analiza, Ni-Pd/Al₂O₃, keramična pena, metoda aerosola

1 INTRODUCTION

While metal-processing technologies can have significant impact on human health, the synthesis of metal/ceramic catalysts is conducted in a laboratory environment with a small risk on human health.¹ Composite metal/ceramic catalysts are used in a wide range of heterogeneous catalytic processes, including reforming of hydrocarbons. Hydrocarbons are reformed in order to obtain H₂ or a synthesis gas (CO+H₂), which are highly efficient energy sources.^{2,3} The most common catalysts for this purpose are based on the Ni/Al₂O₃ system.^{4,5} Despite high catalytic properties of noble-metal-based catalysts, their application is not economically favorable in industrial conditions.^{6,7} Ni is an effective alternative for noble metals due to its low price, good catalytic activity and selectivity. In order to prevent a deactivation of active Ni particles, a low amount of an activity modifier, such as 0.1 % of mass fractions of Pd, is added.^{2,3}

Conventional thermochemical methods for the Ni/Al₂O₃ catalyst synthesis are impregnation, precipitation, coprecipitation and others. Oxide precursors for Ni that are supported by an Al₂O₃ powder are obtained from aqueous solutions of nickel salts and calcination in air. Also, both Al₂O₃ and oxide precursors for Ni can be prepared from the aqueous solutions of the corresponding metal salts. Calcination is carried out to form mixed oxides. Catalysts are then obtained with a hydrogen reduction and they are usually in the form of a powder. During the calcination procedure, undesirable and hardly reducible NiAl₂O₄ is commonly formed. In order to reduce this phase to Ni, high reduction temperatures are required.^{4,8,9}

There are novel methods for the catalyst synthesis, which include aerosol generation in order to form the precursors for active metals. In a previous research¹⁰, authors synthesized a monolithic Ni-Pd/Al₂O₃ catalyst supported by a Al₂O₃-based foam, using a novel method. The foam was prepared according to the previously

presented procedure¹¹ and then impregnated with ultrasonically generated aerosols of chlorides of Ni and Pd. The calcination step was eliminated and the catalyst was reduced at a very low temperature, which allowed a simplification of the synthesis process and energy savings. The obtained catalyst was intended for dry methane reforming, where the synthesis gas is produced from CH₄ and CO₂.^{10,12}

Considering the work of the previous authors on the catalyst-synthesis methods, it can be concluded that none of the studies compared the catalyst-synthesis methods using a multi-criteria analysis. In this research, the novel synthesis method for the Ni-Pd/Al₂O₃ catalyst is compared with the other Ni-based catalyst-synthesis methods using three multi-criteria analysis methods.

2 MATERIALS AND METHODS

Parameters (criteria) for assessing the catalyst-synthesis methods are:

P1 – Preparation method [-], expressed with a five-point scale. Impregnation is carried out by soaking the Al₂O₃ powder in a Ni(NO₃)₂ aqueous solution. With respect to the precipitation, the Al₂O₃ powder is immersed into the Ni(NO₃)₂ solution and a precipitation agent is added. Coprecipitation involves mixing the Al(NO₃)₃ solution (this salt is the precursor for Al₂O₃) and the Ni(NO₃)₂ solution, and then the precipitation agent is added.

P2 – Addition of the precipitation agent during the preparation [-], expressed with a two-point scale: without the addition – 0 points, with the addition – 1 point. Impregnation is used to deposit Ni ions to the Al₂O₃ surface. During the precipitation, the initial precursor for Ni is deposited into Al₂O₃ from the salt solution, by increasing its pH value with the precipitation agent. During the coprecipitation, the initial precursors for both Ni and Al₂O₃ are formed by increasing the pH value of the mixed salt solution.

P3 – Forming and mixing the precursors [Kh], expressed by temperature (K) × time (h). The conventional thermochemical routes require a long time. The mixing of appropriate solutions with the Al₂O₃ powder during the impregnation and precipitation is usually performed at 293 K for 24 h. During the coprecipitation, solutions are stirred between 313–358 K for 1–10 h.

P4 – Filtration [-], expressed with a five-point scale. The solid deposit and the liquid phase are separated with filtration. After the impregnation, the deposit is only filtered and dried in an oven. However, after the precipitation and coprecipitation, the deposit is first filtered, dried and rinsed, usually with distilled water. Then, the drying procedure is repeated.

P5 – Drying procedure [Kh], expressed with temperature (K) × time (h). The drying of the solid deposit is carried out between 353–383 K for 10 h.

P6 – Calcination parameters [Kh], expressed with temperature (K) × time (h). Dried deposits are calcined in air atmosphere, commonly between 873–973 K for 3–6 h, to prepare mixed oxides.

P7 – Reduction parameters [Kh], expressed with temperature (K) × time (h). To activate the catalysts, mixed oxides are reduced by H₂, usually between 873–973 K for 1–3 h. Metallic Ni is obtained from an oxide precursor and Al₂O₃ remains as the oxide.

P8 – Presence of NiAl₂O₄ [-], expressed with a two-point scale: not present – 0 points, present – 1 point. The NiAl₂O₄ spinel phase, which occurs during the calcination, is highly undesirable due to a low reducibility. Its presence causes a lower amount of the active metallic Ni and lower catalyst performances.

The considered catalyst-synthesis methods are:

CSM1 – Ni/Al₂O₃, 15 % Ni – impregnation – Powder γ -Al₂O₃ and aqueous Ni(NO₃)₂ solution were stirred at room temperature for 24 h and filtered. The deposit was dried at 383 K for 10 h and calcined at 873 K for 3 h. Reduction was performed at 873 K for 2 h.⁴

CSM2 – Ni/Al₂O₃, 15 % Ni – precipitation – The Ni(NO₃)₂ solution was added to the solution of the precipitating agent (Na₂CO₃), which contained the γ -Al₂O₃ powder. After stirring the mixture for 24 h, the deposit was filtered, dried at 353 K for 10 h, then rinsed with distilled water and dried again at 383 K for 10 h. The obtained deposit was calcined at 873 K for 3 h and reduced at 873 K for 2 h.⁴

CSM3 – Ni/Al₂O₃, 15 % Ni – coprecipitation – A solution of Ni(NO₃)₂ and Al(NO₃)₃ was added drop-wise into the Na₂CO₃ solution. After constant stirring at 313 K, the resulting deposit was additionally stirred for 1 h. The filtered deposit was dried at 353 K for 10 h, rinsed with distilled water and then dried at 383 K for 10 h. Calcination parameters were 873 K and 3 h. Reduction was performed at 873 K for 2 h.⁴

CSM4 – Ni/Al₂O₃, 20 % Ni – sequential precipitation – Separate solutions of Ni(NO₃)₂ and Al(NO₃)₃ were prepared. First, a NH₄OH solution was added to the Al(NO₃)₃ solution in order to form a white deposit. Then, the NH₄OH and Ni(NO₃)₂ solutions were slowly added to the solution with the white deposit until a blue deposit was obtained. The resulting product was kept at 293 K for 24 h. The separation of the deposit from the liquid was followed by rinsing with ethanol, drying at 373 K for 10 h, calcination at 973 K for 5 h and reduction at 973 K for 3 h.¹³

CSM5 – Ni/Al₂O₃, 50 % Ni – coprecipitation – Dissolved Ni(NO₃)₂ and Al(NO₃)₃ were mixed with urea (the precipitation agent) and stirred at 358 K for 10 h. The deposit was washed, filtered and dried at 383 K for 10 h. Calcination was carried out at 923 K for 6 h and the catalyst was reduced at 973 K for 1 h.⁹

CSM6 – Ni/Al₂O₃, 50 % Ni – coprecipitation – This catalyst was prepared according to the *CSM5* procedure,

Table 1: Performance matrix: properties of synthesis methods for Ni-based catalysts

Tabela 1: Matrika uspešnosti: lastnosti metod sinteze katalizatorjev na osnovi Ni

Parameter/ catalyst	P1				P2	P3	P4				P5	P6	P7	P8
	DM1	DM2	DM3	Sum			DM1	DM2	DM3	Sum				
CSM1	3	2	3	8	0	7032	3	3	3	9	3830	2619	1746	1
CSM2	4	3	5	12	1	7032	1	1	2	4	7360	2619	1746	0
CSM3	2	1	1	4	1	313	1	1	2	4	7360	2619	1746	1
CSM4	4	3	5	12	1	7032	3	2	3	8	3730	4865	2919	1
CSM5	2	1	1	4	1	3580	3	2	3	8	3830	5538	973	0
CSM6	2	1	1	4	1	3580	3	2	3	8	3830	5538	973	1
CSM7	3	5	4	12	0	473	5	5	5	15	473	0	799,5	0

with the same calcination and reduction conditions. K_2CO_3 was used as the precipitation agent.⁹

CSM7 – Ni-Pd/Al₂O₃, 20 % Ni, 0.1 % Pd – The authors synthesized this catalyst with a novel method, that involved an impregnation of Al₂O₃-based foams with an ultrasonically generated aerosol of chlorides of Ni and Pd. Impregnation was performed for 1 h at 473 K. Afterwards, the chlorides deposited onto the foam were dried at 473 K for 1 h. Calcination was eliminated and the reduction of the chlorides was carried out for 1.5 h at a very low temperature – 533 K. The chlorides were nearly completely reduced and their reduction degree was 98.2 % of mass fractions.¹⁰

The performance matrix, the properties of the catalysts based on system Ni/Al₂O₃ synthesized with different methods, is given in **Table 1**. Parameters P1 and P4 represent the decision makers' preference according to their experience, expressed with points in the range of one to five. The final parameter values for P1 and P4 are obtained as the sum of all decision makers' preferences. The five-point rating scale was used for evaluating parameters P1 and P4, where one point represents the least desirable and five points represent the most desirable solution.

SAW (Simple Additive Weighting), TOPSIS (Technique for Order Preference by Similarity to Ideal Solution) and PROMETHEE II (Preference Ranking Organization METHod for Enrichment Evaluation) methods were used for a multi-criteria assessment of the catalyst-synthesis methods.

SAW is a simple method that usually provides results similar to the ones obtained with more complex methods.¹⁴ After assigning the parameter weighting factors w_j using Equation (1) for every alternative, the total value S_i is calculated, where the best alternative has the largest S_i value:

$$S_i = \sum_{j=1}^m w_j b_{ij}; i=1,2,\dots,n \quad (1)$$

$$\sum_{j=1}^m w_j = 1; \quad 0 \leq w_j \leq 1$$

Normalized performance-matrix elements b_{ij} are calculated with Equations (2) and (3):

$$b_{ij} = \frac{a_{ij} - a_j^-}{a_j^* - a_j^-} \quad (2)$$

for the parameters that have to be maximized,

$$b_{ij} = 1 - \frac{a_{ij} - a_j^-}{a_j^* - a_j^-} \quad (3)$$

for the parameters that have to be minimized,

where: S_i – rating of i th-alternative (alternative rank), w_j – parameter weighting factor, b_{ij} – normalized matrix element, a_{ij} – performance matrix element, a_j^* – j -parameter ideal solution (the maximum value), a_j^- – j -parameter anti-ideal solution (the minimum value), n – number of alternatives, m – number of parameters.

The basic principle of the TOPSIS method is that the chosen alternative should have the shortest distance from the ideal solution and the farthest distance from the negative-ideal solution.¹⁵ The TOPSIS procedure consists of six steps:

1. *Normalization of the performance matrix.* The performance matrix $X_{(n,m)}$, where each matrix row corresponds to one alternative, and each column to a single criterion, is normalized with the following Equation (4):

$$r_{ij} = \frac{x_{ij}}{\sqrt{\sum_{i=1}^n x_{ij}^2}} \quad i = 1, \dots, n, j = 1, \dots, m \quad (4)$$

2. *Calculation of the weighted normalized performance matrix.* Weighted normalized performance-matrix values v_{ij} are calculated in Equation (5) as:

$$v_{ij} = w_j r_{ij} \quad (5)$$

where w_j is the parameter weight and $\sum w_j = 1$.

3. *Determining the ideal and anti-ideal solutions.* The ideal solution A^+ and anti-ideal solution A^- are determined with Equations (6) and (7):

$$A^+ = \{(\max v_{ij} | j \in G), (\min v_{ij} | j \in G^c), i=1, \dots, n\} = \{v_1^+, v_2^+, \dots, v_m^+\} \quad (6)$$

$$A^- = \{(\min v_{ij} | j \in G), (\max v_{ij} | j \in G^c), i=1, \dots, n\} = \{v_1^-, v_2^-, \dots, v_m^-\} \quad (7)$$

where:

$G = \{j = 1, 2, \dots, m \mid j \text{ parameters that have to be maximized}\}$

$G' = \{j = 1, 2, \dots, m \mid j \text{ parameters that have to be minimized}\}$

4. Calculations of the distances from the ideal and anti-ideal solutions.

$$D_i^+ = \sqrt{\sum_{j=1}^m (v_{ij} - v_j^+)^2}, i=1, \dots, n \quad (8)$$

$$D_i^- = \sqrt{\sum_{j=1}^m (v_{ij} - v_j^-)^2}, i=1, \dots, n \quad (9)$$

5. Calculation of relative closeness (RC) to ideal solution.

$$RC_i^+ = \frac{D_i^-}{D_i^+ + D_i^-}, i = 1, \dots, n \quad (10)$$

where $0 \leq RC_i^+ \leq 1$.

6. Ranking of alternatives. Alternatives are ranked by descending values of RC_i^+ .

The PROMETHEE I method provides a partial ranking, while PROMETHEE II provides a complete ranking of alternatives.^{16,17} The main feature of the PROMETHEE method is the fact that for each parameter, the decision maker assigns one of the six predefined preference functions provided in ^{16,17}. Only the two preference functions, which are used in this paper, are described here. The usual preference function (Equations (11) and (12)) is used for qualitative criteria (P1 and P4), while a V-shape preference function (Equations (13) and (14)) is used for quantitative criteria (P2, P3, P5, P6, P7, and P8):

$$P_{\text{usual}}(a, b) = \begin{cases} 0, & d(a, b) \leq 0 \\ 1, & d(a, b) > 0 \end{cases} \quad (11)$$

parameters that have to be maximized

$$P_{\text{usual}}(a, b) = \begin{cases} 0, & d(a, b) > 0 \\ 1, & d(a, b) \leq 0 \end{cases} \quad (12)$$

parameters that have to be minimized

$$P_{\text{V-shape}}(a, b) = \begin{cases} \frac{|d(a, b)|}{p}, & d(a, b) \leq p \\ 1, & d(a, b) > p \end{cases} \quad (13)$$

parameters that have to be maximized

$$P_{\text{V-shape}}(a, b) = \begin{cases} \frac{|d(a, b)|}{p}, & d(a, b) > p \\ 1, & d(a, b) \leq p \end{cases} \quad (14)$$

parameters that have to be minimized

where $d_j(a, b)$ is a deviation in the assessment of alternative a over alternative b for each parameter, and p is the preference threshold.

PROMETHEE II can be calculated through the following five steps:

1. Defining the deviations according to pairwise comparisons,

$$d_j(a, b) = g_j(a) - g_j(b) \quad (15)$$

where $d_j(a, b)$ is the deviation in the assessment of alternative a over alternative b for each parameter.

2. Applying the preference functions,

$$P_j(a, b) = F_j[d_j(a, b)]; j=1, 2, \dots, m \quad (16)$$

where $P_j(a, b)$ is the preference of alternative a over alternative b for each parameter, as function $d_j(a, b)$.

3. Calculating the total preference index,

$$\forall a, b \in A, \pi(a, b) = \sum_{j=1}^m P_j(a, b)w_j \quad (17)$$

where $\pi_j(a, b)$, from a to b (from 0 to 1), is defined with the weighted sum $P_j(a, b)$ for each j -parameter, and w_j is the weighting factor of j -parameter.

4. Calculating the positive (ϕ^+) and negative flows (ϕ^-) – PROMETHEE I partial ranking,

$$\phi^+(a) = \frac{1}{n-1} \sum_{x \in A} \pi(a, x) \quad (18)$$

$$\phi^-(a) = \frac{1}{n-1} \sum_{x \in A} \pi(x, a) \quad (19)$$

5. Calculating the net flow (ϕ) – PROMETHEE II complete ranking,

$$\phi(a) = \phi^+(a) - \phi^-(a) \quad (20)$$

Parameter preferences of the decision makers (parameter weight, type, PROMETHEE preference function and preference threshold) for MCA are provided in **Table 2**. Parameter weighting factors (in this case DM1, DM2 and DM3) are assigned by the decision makers, who are experts in field of Nickel-based catalysts, for

Table 2: Parameter preferences

Tabela 2: Nastavitve parametrov

Parameter preferences	P1	P2	P3	P4	P5	P6	P7	P8
Parameter weight (DM1)	1	2	3	2	6	9	7	6
Parameter weight (DM2)	2	2	6	2	8	10	9	6
Parameter weight (DM3)	1	1	5	1	5	9	6	8
Final parameter weight	0.0333	0.0426	0.1204	0.0426	0.1611	0.2407	0.1870	0.1741
Parameter type	Max	Min	Min	Max	Min	Min	Min	Min
PROMETHEE Pref. function	Usual	Usual	V-shape	Usual	V-shape	V-shape	V-shape	Usual
PROMETHEE Pref. threshold	–	–	6000	–	6000	5000	2000	–

each parameter on a ten-point scale. The final, aggregated weighting factor is calculated with the following Equation (21):

$$w_{F,j} = \frac{1}{k} \sum_{k=1}^l \frac{w_{DM,k,j}}{\sum_{j=1}^m w_{DM,k,j}}, j=1, \dots, m; k=1, \dots, l \quad (21)$$

where $w_{F,j}$ is the final weighting factor, $w_{DM,k}$ is the weighting factor of k -th decision maker, m is the number of parameters, l is the number of decision makers.

3 RESULTS AND DISCUSSION

According to the decision makers' preferences (Table 2), parameters P6, P7, P5, and P8 were considered as the most important. The qualitative parameters, obtained on the basis of the decision makers' preferences on the five-point scale, P1 and P4, were considered as the least important by the decision makers. After the decision makers expressed their preferences about the parameters for the catalyst-synthesis methods from Table 2, three MCA methods (SAW, TOPSIS, and PROMETHEE II) were applied in order to obtain the results shown in Figures 1 and 2.

The normalization results of the TOPSIS method (vector normalization), shown in Figure 1, show the "default scenario" without the decision makers' preferences. From Figure 1 it is clear that catalyst Ni-Pd/Al₂O₃ (CSM7) stands out from the rest. The normalization results for catalyst Ni-Pd/Al₂O₃ are affected by the fact that in the performance matrix (Table 1) catalyst Ni-Pd/Al₂O₃ has the best values (ideal solution) for parameters P2, P4, P5, P6 and P7.

As in the normalization results, the results for all three MCA methods (SAW, TOPSIS, PROMETHEE II) shown in Figure 2, indicate that the best ranked catalyst is Ni-Pd/Al₂O₃. When the decision makers' weighting factors are included in the three MCA methods (Figure 2), Ni-Pd/Al₂O₃ is even more distinguished from the other catalysts than in the normalization results (Figure

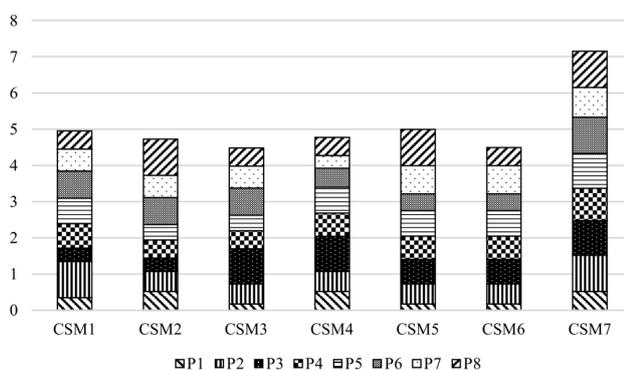


Figure 1: Normalization results (vector normalization) in TOPSIS
Slika 1: Rezultati normalizacije (vektorska normalizacija TOPSIS)

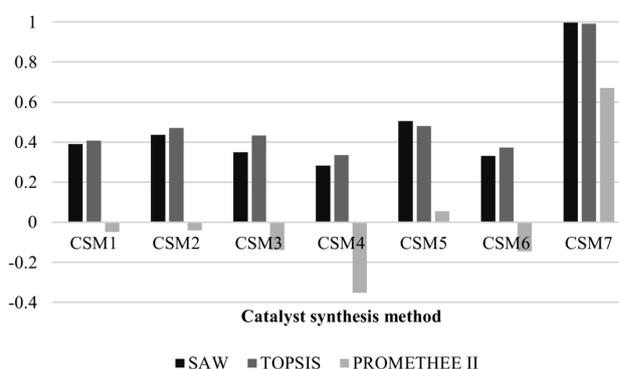


Figure 2: Ranking of catalyst-synthesis methods
Slika 2: Razvrstitev metod sinteze katalizatorja

1). On the other hand, catalyst-synthesis methods CSM3, CSM4 and CSM6 are ranked as the poorest.

4 CONCLUSION

The best ranked catalyst-synthesis method is the novel method for obtaining the Ni-Pd/Al₂O₃ catalyst according to the normalization results (Figure 1) and the results for all three MCA methods (SAW, TOPSIS, PROMETHEE II) (Figure 2). For a multi-criteria analysis, it is common that the results of different methods differ to some extent, as in this case. However, all three methods indicated the Ni-Pd/Al₂O₃ catalyst as the best solution, which is a strong proof that the novel catalyst-synthesis method stands out from the rest.

The Ni-Pd/Al₂O₃ catalyst was synthesized with a novel method, which includes impregnation of Al₂O₃ based foams with an ultrasonically generated aerosol of metal chlorides. The method enabled elimination of the calcination step and the chloride precursors were almost completely reduced (98.2 % of mass fractions) at a very low temperature of 533 K. This may provide economic and technological benefits in the catalyst production process.

Future research will be focused on the life-cycle analysis of the synthesis method for the Ni-Pd/Al₂O₃ catalyst, where the consumption of natural resources and energy sources in the production phase will be analyzed. Assessment results for the life-cycle impact will provide more information about the synthesis method for the Ni-Pd/Al₂O₃ catalyst and environmental impacts on impact categories such as human health, natural resources and ecosystem quality.

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