PROSPECTS FOR CREATING INDICATOR SYSTEMS BASED ON CERAMIC MATERIALS

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The article presents the prospects for creating indicator systems based on ceramic membranes for field use. The aim of this work is to analyse the prospects and current approaches to creating indicator systems based on ceramic membranes modified with organic reagents, with an emphasis on detecting nitrogen-containing compounds and heavy metal ions in aqueous environments. It is noted that in order to create indicator ceramic membranes with high stability, sensitivity and selectivity, their preliminary modification is necessary. An analysis of methods for modifying ceramic materials indicates that covalent bonding is the most promising, but modifying membranes by polymer coating is more promising where high concentrations of analytes are required. Despite its simplicity, the direct adsorption method is not promising for the creation of long-term indicator membranes due to their low stability. It has been found that for the qualitative determination of nitro compounds, it is reasonable to use a simple method involving the use of iron(II) ions, which combines low cost, simplicity, stability and the possibility of effective immobilisation on ceramic materials. For the qualitative and quantitative determination of heavy metal ions, the creation of indicator membranes based on azo dyes, in particular chromazurol S, is promising. In this case, silicon(IV) oxide is the optimal ceramic carrier due to its high specific surface area and reactivity, which, in combination with modification by the polymer layer method, ensures the highest dye content, and by covalent bonding, the best stability. Thus, the article presents great prospects for the creation of indicator systems based on ceramic materials for obtaining indicator systems for field conditions.

Keywords: *ceramic membranes, environmental monitoring, heavy metals, indicator systems, nitrogen-containing compounds, silanisation, surface modification*

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1. Introduction

The active growth of risks associated with the use of explosive substances in military and terrorist acts creates an urgent need to develop rapid, economical and safe methods for detecting them in external environments. Monitoring water bodies is particularly important, as it is through them that explosive nitroorganic compounds and heavy metals can spread over long distances, enter drinking water reservoirs and cause damage to ecosystems. Controlling the content of such pollutants is crucial from both an environmental and security perspective, especially given the lack of a comprehensive environmental monitoring system in many regions of Ukraine (Sharma et al., 2025; Zamora-Ledezma et al., 2021).

Modern analytical chemistry offers a number of methods for determining toxic compounds, but most of them require complex equipment and highly qualified personnel. This limits their application directly in the field. Against this background, qualitative methods with visual indication based on a change in the colour of the reaction medium and which can be integrated into portable sensor systems are of particular interest (US EPA, 2025).

Indicator systems are widely used in analytical chemistry and environmental monitoring and can exist in various forms: drop solutions, test strips, powder forms, and electrode systems with membranes. Commercial samples of indicator systems are often based on ceramic materials, in particular silica gels or Al_2O_3, but their use in powder form has significant limitations – difficulty in dosing, uneven distribution of reagents, susceptibility to agglomeration, and storage problems (Namieśnik, 2000).

Ceramic membranes, which provide controlled porosity, stable active surface geometry, and precise fixation of analytical reagents, have been a significant step forward in this field (Molchan et al., 2025). The presence of hydroxyl groups, Lewis and Brønsted-Lowry acid and base centres on the surface of such materials determines their high reactivity. Due to this, ceramic membranes remain effective even in aggressive environments, which opens up wide opportunities for the creation of stable and durable sensor systems (Malebadi et al., 2025).

Among ceramic materials, oxide systems based on Al_2O_3 and TiO_2 are of greatest interest, as they combine high specific surface area, chemical stability, and the possibility of targeted modification of active centres. Such properties make them particularly promising for the development of indicator membranes with high sensitivity and selectivity (Malebadi et al., 2025).

Current research in the field of ceramic membranes is directed not only towards improving their mechanical characteristics, but also towards imparting specific functional properties through chemical modification of the surface with various reagents. Native ceramics exhibit limited selectivity; therefore, functionalisation of their surface with organic analytical reagents allows the combination of stability and strength of the inorganic matrix with the high selectivity and sensitivity of organic complexing agents (Meder and Rezwan, 2022).

Thus, modification of ceramic material surfaces with specific organic reagents creates the foundation for developing indicator systems with high stability, sensitivity, and selectivity. Such systems combine ease of use and the possibility of direct visual indication with the reliability of the ceramic matrix, making them promising for portable technologies in environmental monitoring and water quality control (Meder and Rezwan, 2022).

The aim of this work is to analyse the prospects and current approaches to developing indicator systems based on ceramic membranes modified with organic reagents, with emphasis on the detection of nitrogen-containing compounds and heavy metal ions in aqueous media.

2. Methods of modifying ceramic materials to create indicator systems

The direct adsorption method is based on physical adsorption and electrostatic interaction between functional groups of the dye and the surface of the ceramic material (Etienne, 2003). Under optimal conditions, this method allows the achievement of adsorption capacity up to 42.5 mg/g, which corresponds to approximately 70% surface coverage. The main advantage of the method is its simplicity of implementation and the absence of need for additional reagents (Wang and Chen, 2009).

However, the method is characterised by significant disadvantages in the form of instability of the resulting materials in aqueous solutions, particularly upon changes in environmental pH. For example, the desorption constant of chromazurol S adsorbed on aluminium oxide is 0.012 min^{-1} at pH 7.0 and increases to 0.085 min^{-1} at pH 9.0, rendering such materials unsuitable for prolonged use in analytical applications (Martinez et al., 2006).

Covalent binding of azo dyes to the ceramic carrier surface occurs through silane groups, providing significantly higher stability of the modified materials. The method involves surface functionalisation with a silanising agent, for example 3-aminopropyltriethoxysilane (APTES) and 3-glycidyloxypropyltrimethoxysilane (GPTMS), followed by covalent binding of analytes through the introduced functional groups (Etienne, 2003). When amino groups are used as linkers, the desorption constant typically decreases compared to direct adsorption (Shen et al., 2013). The use of epoxide groups also promotes increased adsorption constant whilst maintaining excellent stability. The disadvantage of this method is the complexity of synthesis and the necessity for anhydrous conditions and specialised reagents, which increases the cost of obtaining modified materials (Kumar et al., 2013).

Formation of a thin polymeric layer on the ceramic carrier surface with subsequent immobilisation of analytes is also a promising approach for increasing the adsorption constant whilst maintaining excellent hydrolytic stability (Etienne, 2003; Zhang and Bai, 2003). For example, the use of polyacrylamide as an intermediate layer ensures uniform distribution of analytes and creates additional binding sites. Alternative application of biocompatible chitosan allows modification to be conducted under milder conditions. The main limitation of the method is the complexity of multistage synthesis and the need for careful control of polymerisation conditions (Kyrii, 2023; Wang et al., 2015).

The layer-by-layer (LbL) method is based on alternating adsorption of oppositely charged polyelectrolytes with formation of a multilayer structure (Etienne, 2003). It has been established that with an increase in the number of bilayers, the content of analytes on the surface can increase linearly (Kaur et al., 2020). The advantages of the method are precise control of modifier layer thickness, uniform dye distribution, and high stability due to multiple electrostatic interactions between layers. However, the method is labour-intensive due to the need to repeat the application cycles multiple times (Etienne, 2003).

Analysis of the presented methods shows that for creating stable indicator systems based on ceramic membranes, modification through silane groups is the most promising approach. This approach provides an optimal balance between material stability, high concentration of immobilised analytes, and synthesis reproducibility.

Among silanising agents, the use of APTES is recommended due to its commercial availability, relatively simple functionalisation methodology, and capability to form stable amide bonds. For specific applications where maximum analyte concentrations are required, modification through a polymer layer may be considered; however, the complexity of synthesis makes this method less attractive for mass production of indicator systems. The direct adsorption method, despite its simplicity, is not recommended for creating long-term indicator systems due to low stability in aqueous solutions under pH variations.

3. Analysis of analytes for the qualitative determination of nitrogen-containing organic compounds

Modern methods of qualitative analysis of organic compounds are increasingly focused on combining classical chemical reactions with materials science approaches, which enables the creation of portable sensor systems. An important group of such systems comprises ceramic matrices modified with organic reagents that provide selective detection of specific pollutants. In the case of nitrogen-containing compounds, the question of selecting adequate analytes is of particular significance, as the selectivity, economic feasibility, and longevity of sensors depend on this choice (Meder and Rezwan, 2022).

In this context, it is advisable to compare different analytes used for the detection of amides, nitriles, amines, and nitro compounds according to three key criteria: cost and availability of reagents, analytical efficiency of the reaction, and potential for impregnation onto ceramic matrices.

For the qualitative determination of amides and nitriles, the classic and most common reagent is hydroxylamine (NH_2OH). It reacts with amide or nitrile groups to form hydroxamic acids or amidoximes, respectively. These products then allow stable and intensely coloured complexes with Fe^{3+} to be obtained, usually red-violet in colour. This reaction is highly selective and the colour changes are quite pronounced, allowing it to be used as the basis for indicator systems. From an economic point of view, hydroxylamine is a relatively cheap and readily available reagent, as evidenced by its widespread use in classical analytical chemistry. An additional advantage is the ease of impregnation onto porous ceramics, where it can be fixed in the form of immobilised solutions or by adsorption (Ahluwalia and Dhingra, 2001).

An alternative approach involves the use of soda lime in combination with $CuSO_4$ solution in methanol. In this case, amides release ammonia upon heating, which reacts with Cu^{2+} to form a blue-violet precipitate. However, this method is less convenient as it requires preliminary heating and is more complex to implement under field detection conditions. Furthermore, reagent impregnation onto the matrix is technically more challenging, whilst the reagents themselves are more expensive and less stable during prolonged use (Trofimenko and Sease, 1958).

Therefore, taking into account economic and technological factors, it can be stated that the most promising method for determining amides and nitriles is the application of the "hydroxylamine–Fe^{3+}" system. It combines reagent accessibility, adequate sensitivity, and simplicity of implementation on ceramic carriers, making it optimal for creating indicator systems.

There is a wide range of analytes available for detecting amines, but their effectiveness and prospects vary considerably. One of the simplest and most accessible reagents is methyl orange. It forms stable yellow complexes with amines in a weakly acidic environment, with the intensity of the colour being directly proportional to the concentration of the analyte. The method is extremely simple, inexpensive, and well suited for immobilisation on ceramic matrices. Methyl orange is widely used in acid-base indicators, so its cost is low, and its availability is high. All these factors make it one of the most promising reagents for practical sensor use (Lovibond, 2025).

Specific to tertiary amines is Dragendorff's reagent (potassium iodobismuthate), which forms brightly coloured complexes with amines and quaternary ammonium salts. However, its use is limited: the reagent is less convenient to apply to the matrix, may lose its activity during prolonged storage, and is also more expensive than methyl orange. Nevertheless, in cases where it is necessary to detect tertiary amines specifically, Dragendorff's reagent retains its importance (Večeřa and Gasparič, 1971).

In modern research, Tinuvin 1130 is also used to detect amines, which allows different colours to be obtained depending on the type of amine (primary, secondary or tertiary). This makes it possible to perform a more selective analysis, but the cost of Tinuvin 1130 is very high, which significantly limits its practical application. In addition, it is less stable during the impregnation process on the matrix. From an economic point of view, its use is unjustified for mass indicator systems, although it is of interest for laboratory research (Palliardi et al., 2022).

Therefore, methyl orange is the most promising analyte for amines, while Tinuvin 1130 should be considered as an additional selective analyte in studies, and Dragendorff's reagent as a specific agent for tertiary amines.

Nitro compounds are challenging analytical targets, as they are characterised by high chemical stability and low reactivity. Several classical methods are employed for their detection. One of the most well-known is the Mulliken-Barker test, which is based on the reduction of the nitro group to hydroxylamine, followed by reduction of Tollens' reagent with formation of a silver precipitate. This method demonstrates good selectivity and sensitivity; however, it has several disadvantages: Tollens' reagent is unstable, difficult to impregnate onto ceramic matrices, and requires freshly prepared solutions. This significantly limits its practical prospects (Ahluwalia and Dhingra, 2001).

A more accessible and convenient method is the reaction with Fe^{2+} ions, which are oxidised by nitro groups to Fe^{3+}, accompanied by the appearance of a characteristic colour and the precipitation of $Fe(OH)_3$. This method is cheaper, easier to perform, and, most importantly, can be easily implemented in the form of impregnated ceramic sensors. From an economic point of view, the use of Fe^{2+} salts is most expedient, as they are cheap and stable in storage (Ahluwalia and Dhingra, 2001).

NaH in acetone is also employed for determining aromatic nitro compounds, providing extremely high sensitivity and rapid colour changes even at microconcentrations (less than 1 μg). However, the extreme reactivity of NaH makes it difficult to use practically in impregnated matrices, whilst the classification of acetone as a 'precursor' renders this method technologically unfeasible. Furthermore, NaH requires special storage conditions, is expensive and hazardous to handle; therefore, its prospects for application in indicator systems are low (Pearson and Morrissey, 1962).

Considering the above factors, the most promising method for the qualitative determination of nitro compounds is the use of Fe^{2+}, which combines low cost, simplicity, stability, and the possibility of effective immobilisation on ceramic materials. However, an analysis of the prospects of various analytes leads to the conclusion that the most effective methods for creating indicator systems based on ceramic membranes are those that combine the economic affordability of reagents with high analytical efficiency and technological compatibility with ceramic carriers. For amides and nitriles, the optimal method is colorimetry with $FeCl_3$, for amines – the method with methyl orange, and for nitro compounds – testing with iron hydroxide as the most balanced solution according to all evaluation criteria. Overall, these analytes provide an optimal balance of economic feasibility, analytical efficiency, and technological convenience for the creation of modern indicator systems for environmental monitoring.

4. Analytes for ceramic indicator materials and determination of heavy metals

For qualitative and quantitative determination of heavy metal ions, various organic reagents are traditionally used – the so-called metallochromic indicators, which form coloured complexes with metal cations. Among the most common are dithizone, used to determine Cu^{2+}, Pb^{2+} and Cd^{2+} ions; xylenol orange, which is sensitive to Zn^{2+}, Cu^{2+}, Cd^{2+}, Pb^{2+}, Mn^{2+} and Fe^{3+}; arsenazo III, which shows high selectivity for Pb^{2+}; as well as various azo dyes, dithiocarbamates and oxime reagents, which are employed for spectrophotometric determination of metals (Abdelmonem et al., 2025). Among these reagents, azo dyes occupy a special place, in particular chromazurol S, which combines versatility with high sensitivity to a wide range of metal ions, including Cu^{2+},Al^{3+} and Fe^{3+}. It forms intensely coloured complexes suitable for both spectrophotometric measurements and visual assessment (Patel et al., 2018). An important advantage of chromazurol S is its low detection limit and stability when impregnated into solid substrates, which enables its effective use in indicator ceramic membranes. These properties make it one of the most promising analytes for the further development of indicator systems for environmental monitoring (Santarossa and Fernández, 2017).

An analysis of modern approaches to modifying ceramic materials using chromazurol S has identified four main methods: direct adsorption, covalent bonding through silane groups, modification through a polymer layer, and the LbL method (Etienne, 2003). Comparative characteristics of these methods are presented in Table 1 (Wang and Chen, 2009; Martinez et al., 2006; Shen et al., 2013; Kumar et al., 2013; Zhang and Bai, 2003; Wang et al., 2015; Kaur et al., 2020).

As can be seen from Table 1, the highest content of immobilised chromazurol S is achieved when using a polymer layer, which is explained by a significantly larger number of functional groups compared with an unmodified surface. The most stable materials are formed through covalent bonding and modification via the polymer layer owing to the formation of strong covalent bonds.

Notably, although the direct adsorption method is characterised by the simplest technique and minimum synthesis time, it provides the lowest stability of modified materials. The LbL method demonstrates the best uniformity of modifier distribution due to the sequential application of thin layers. The nature of the ceramic carrier also significantly affects the effectiveness of modification, as evidenced by the comparative analysis of main ceramic materials using the covalent bonding method, presented in Table 2 (Shen et al., 2013; Kumar et al., 2013).

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Table 1. *Comparative characteristics of methods for modifying ceramic materials with chromazurol S (Wang and Chen, 2009; Martinez et al., 2006; Shen et al., 2013; Kumar et al., 2013; Zhang and Bai, 2003; Wang et al., 2015; Kaur et al., 2020)*

Parameter	Direct adsorption	Covalent bonding through silane groups	Modification through polymer layer	Layer-by-layer method
Maximum content of chromazurol S, mmol/g	0.05-0.10	0.20-0.30	0.40-0.50	0.20-0.30
Desorption constant at pH 9.0, h^{-1}	0.085	<0.001	<0.0005	0.003
Stability in pH range	2-7	2-10	2-11	2-9
Synthesis time, h	6-24	36-48	48-72	10-30
Uniformity of modifier distribution	Low	High	High	Very high

In particular, silica gel has the highest content of immobilised chromazurol S due to its high specific surface area and significant concentration of reactive silanol groups. Aluminium oxide contains a slightly lower content of chromazurol S after modification, despite the higher concentration of OH groups, due to its smaller specific surface area and lower reactivity of the aluminol groups. Titanium and zirconium dioxides are characterised by the lowest dye content due to their limited specific surface area.

Table 2. *Comparative characteristics of ceramic carriers modified with chromazurol S by covalent bonding through silane groups (Shen et al., 2013; Kumar et al., 2013)*

Parameter	Silica gel (SiO_2)	Aluminium oxide (Al_2O_3)	Titanium dioxide (TiO_2)	Zirconium dioxide (ZrO_2)
Specific surface area of starting material, m^2/g	300-600	150-300	50-150	40-100
Concentration of surface OH groups, OH/nm^2	4.6-5.8	8.0-12.0	5.0-7.0	4.0-6.0
Chromazurol S content, mmol/g	0.25-0.30	0.20-0.25	0.10-0.15	0.08-0.12
Chromazurol S binding efficiency, %	30-35	35-40	33-37	30-35

The pH value of the medium is a critical parameter for all modification methods. For example, an acidic medium (pH 3.0-4.0) is optimal for direct adsorption due to the predominance of electrostatic interactions between the positively charged surface and the negative groups of chromazurol S. Covalent bonding achieves maximum efficiency in a neutral medium (pH 6.5-7.5), which provides optimal conditions for carbodiimide activation. The LbL method requires a pH close to neutral (6.0-7.0) for the stability of polyionic complexes (Wang and Chen, 2009; Martinez et al., 2006; Shen et al., 2013; Kumar et al., 2013; Zhang and Bai, 2003; Wang et al., 2015; Kaur et al., 2020). Research into the effect of temperature on silica gel modification through covalent bonding has shown that the optimum temperature range is approximately 60-80°C.

Increasing the temperature to these values increases the silanisation rate and chromazurol S content from 0.18 mmol/g at 25°C to 0.33-0.34 mmol/g at 60-80°C. A further increase in temperature leads to a decrease in efficiency due to partial degradation of the silanising agent (Shen et al., 2013; Kumar et al., 2013). Thus, pH and temperature have a significant effect on the efficiency of covalent binding of analytes to the ceramic matrix.

In conclusion, analysis of methods for modifying ceramic materials with chromazurol S showed that the polymer layer method provides the highest dye content, while covalent bonding provides the best stability. Silica gel is the optimal ceramic carrier due to its high specific surface area and reactivity. The optimal conditions for modifying silica gel with chromazurol S are a temperature of 60-80°C, an APTES concentration of 10%, a silanisation time of 8-12 hours, and a dye immobilisation time of 20-24 hours. The scientific results already available in the literature provide a basis for the development of effective indicator membranes for the determination of heavy metals.

5. Conclusions

This article examines the current state of indicator system utilisation, with particular emphasis on analysing the prospects and modern approaches to creating indicator systems based on ceramic membranes. It is demonstrated that the development of indicator ceramic membranes with high stability, sensitivity, and selectivity requires their prior modification.

The analysis of methods for functionalising ceramic membranes has revealed that the most promising method is covalent bonding, but modifying ceramic materials by coating them with polymer is more promising, as it allows high concentrations of analytes to be fixed to them. It is noted that the direct adsorption method, despite its simplicity, is not recommended for creating long-term indicator systems due to their low stability.

It has been shown that for the qualitative determination of nitro compounds, it is reasonable to use a simple method that mainly involves the use of iron(II) ions and combines low cost, simplicity, stability, and the possibility of effective immobilisation on ceramic materials. In particular, for amides and nitriles, the optimal method is colorimetry with $FeCl_3$, for amines – the method with methyl orange, and for nitro compounds – the method with iron hydroxide.

Analysis reveals that for the purpose of creating indicator membranes for qualitative and quantitative determination of heavy metal ions, azo dyes are the most promising, particularly chromazurol S, which in combination with modification by the polymer layer method provides the highest dye content, whilst covalent bonding provides the best stability. In this case, silicon(IV) oxide is the optimal ceramic carrier due to its high specific surface area and reactivity.

Consequently, analysis of the prospects for utilising different analytes in indicator membrane development leads to the conclusion that the most effective approaches combine economically accessible reagents, high analytical efficiency, strong attachment to ceramic materials, and ease of technological application.

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ПЕРСПЕКТИВИ СТВОРЕННЯ ІНДИКАТОРНИХ СИСТЕМ НА ОСНОВІ КЕРАМІЧНИХ МАТЕРІАЛІВ

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В статті представлено перспективи створення індикаторних систем на основі керамічних мембран для польового використання. Метою даної роботи є аналіз перспектив та сучасних підходів до створення індикаторних систем на основі керамічних мембран, модифікованих органічними реагентами, з акцентом на виявлення нітрогенвмісних сполук та іонів важких металів у водних середовищах. Зазначено, що для створення індикаторних керамічних мембран із високою стабільністю, чутливістю та селективністю необхідне їх попереднє модифікування. Аналіз методів модифікування керамічних матеріалів вказує про найбільшу перспективність ковалентного зв'язування, але модифікування мембран через нашаровування полімеру є більш перспективним, де потрібні високі концентрації аналітів. Метод прямої адсорбції, незважаючи на простоту, не є перспективним для створення довгострокових індикаторних мембран через їх низьку стабільність. Виявлено, що для якісного визначення нітросполук обґрунтовано використання простого методу, який передбачає використання іонів феруму(II), що поєднує дешевизну, простоту, стабільність і можливість ефективної іммобілізації на керамічних матеріалах. Для якісного та кількісного визначення іонів важких металів перспективним є створення індикаторних мембран на основі азобарвників, зокрема хромазуролу S. У цьому випадку силіцій(IV) оксид є оптимальним керамічним носієм завдяки високій питомій поверхні та реакційній здатності, який у поєднанні з модифікуванням за методом полімерного шару забезпечує найвищий вміст барвника, а за ковалентним зв'язуванням – найкращу стабільність. Таким чином, в статті представлені великі перспективи створення індикаторних систем на основі керамічних матеріалів для одержання індикаторних систем для польових умов.

Ключові слова: *важкі метали, екологічний моніторинг, індикаторні системи, керамічні мембрани, модифікування поверхні, нітрогенвмісні сполуки, силанізація*