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DEVELOPMENT OF HERITAGE-COMPATIBLE RESTORATION MORTARS BASED ON LOCAL RAW MATERIALS AND INDUSTRIAL WASTE FOR HISTORICAL MONUMENTS OF UZBEKISTAN

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Annotatsiya. Mazkur tadqiqotda O‘zbekiston tarixiy me’moriy obidalarining g‘isht bilan mos restavratsion kompozit qorishma materiallarni mahalliy xomashyolar va shakar ishlab chiqarish chiqindilari asosida yaratish o‘rganildi. An’anaviy “Hajja” qorishmalarining fizik-kimyoviy xossalarini qayta tiklash maqsadida lyossimon jins, NaCl, $CaCO_3$, Fe_2O_3 hamda defekatdan olingan mikrokalsit asosidagi eksperimental tarkiblar ishlab chiqildi. Materiallar rentgenoflyuorestsent tahlil (XRF), infraqizil spektroskopiya (FTIR), rentgen difraksion tahlil (XRD) va differensial-termik tahlil (DTA/DSC) usullari yordamida o‘rganildi. Olingan natijalar ishlab chiqilgan kompozitsiyalar bilan tarixiy qorishmalar o‘rtasida yuqori mineralogik va strukturaviy moslik mavjudligini ko‘rsatdi. Tadqiq etilgan tarkiblar orasida F-10 namunasi zich mikrostrukturasi va past g‘ovakligi tufayli eng yaxshi natijani namoyon qildi.

Kalit so‘zlar: restavratsion qorishma, “Hajja” qorishmasi, tarixiy g‘isht terimi, mikrokalsit, madaniy merosni saqlash, barqaror restavratsiya

Аннотация. В данной работе исследовано создание реставрационных композитов, совместимых с историческими каменными кладками Узбекистана, с использованием местного сырья и отходов сахарного производства. Для воспроизведения физико-химических свойств традиционных растворов «Hajja» были разработаны экспериментальные составы на основе лёссовидного грунта, NaCl, $CaCO_3$, Fe_2O_3 и микрокальцита, полученного из дефеката. Материалы были исследованы методами рентгенофлуоресцентного анализа (XRF), инфракрасной спектроскопии (FTIR), рентгенодифракционного анализа (XRD) и дифференциально-термического анализа (DTA/DSC). Полученные результаты показали высокую минералогическую и структурную совместимость разработанных композитов с историческими растворами. Среди исследованных составов композиция F-10 продемонстрировала наилучшие свойства благодаря плотной микроструктуре и низкой пористости.

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Ключевые слова: реставрационный раствор, раствор «Hajja», историческая кладка, микрокальцит, сохранение культурного наследия, устойчивая реставрация

Abstract. This study investigates the development of restoration composites compatible with historical masonry structures of Uzbekistan using local raw materials and sugar industry waste. Experimental compositions based on loess-like soil, NaCl, CaCO3, Fe2O3 and defecate-derived microcalcite were developed to reproduce the physicochemical properties of traditional Hajja mortars. The materials were analyzed using XRF, FTIR, XRD and DTA/DSC methods. The obtained results demonstrated high mineralogical and structural compatibility between the developed compositions and historical mortars. Among the investigated compositions, F-10 exhibited the best performance due to its dense microstructure and low porosity.

Keywords: restoration mortar, Hajja mortar, historical masonry, microcalcite, heritage conservation, sustainable restoration

1. INTRODUCTION

Historical architectural monuments represent an important component of the cultural and scientific heritage of humanity. The preservation and restoration of historical masonry structures require the development of restoration materials compatible with the original building systems in terms of mineralogical composition, pore structure, physicochemical stability and durability [1,2]. In many historical buildings, the use of incompatible cement-based repair mortars has resulted in accelerated deterioration processes, formation of internal stresses, cracking and salt migration phenomena [6,7]. Therefore, the development of heritage-compatible restoration mortars remains one of the most important challenges in conservation science and restoration engineering [8].

Traditional restoration mortars used in historical monuments of Central Asia, particularly in the architectural heritage of Uzbekistan, are characterized by complex carbonate–silicate structures formed under long-term environmental воздействия [2]. Studies conducted by Tojiev [2] demonstrated that historical mortars of Uzbek architectural monuments contain significant amounts of quartz, calcite, aluminosilicates and soluble salts, which determine their physicochemical behavior and long-term stability. Similar conclusions regarding the importance of mineralogical compatibility between historical and restoration mortars were also reported by Caroselli et al. [6] and Loke et al. [7].

Lime-based mortars remain among the most widely used materials in restoration practice due to their vapor permeability, carbonation ability and compatibility with historical masonry systems [3,8]. However, pure lime mortars often possess insufficient mechanical strength and low resistance to aggressive

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environmental conditions [4]. According to Gameiro et al. [3], the incorporation of pozzolanic additives into lime mortars significantly improves their carbonation kinetics, structural densification and durability characteristics. Wang et al. [4] further demonstrated that metakaolin-modified hydraulic lime mortars exhibit enhanced compressive strength and reduced porosity compared to conventional lime systems.

Recent studies have focused on the development of sustainable restoration materials based on industrial by-products and waste-derived mineral additives [5,9]. Zhao et al. [5] reported that artificial hydraulic lime composites containing carbonate-rich additives demonstrate improved structural stability and long-term performance in restoration applications. Repina et al. [9] developed cementless restoration compositions based on lime binders and silicon dioxide, emphasizing the importance of environmentally friendly restoration technologies.

One of the promising directions in sustainable materials science is the utilization of sugar industry waste, particularly defecate-derived microcalcite, as a mineral filler and carbonate component for restoration mortars [1]. Defecate waste contains high concentrations of calcium carbonate and can contribute to pore filling, structural stabilization and enhancement of adhesion properties in lime-based composites [1]. The use of such industrial waste also supports circular economy principles and reduces the environmental impact associated with waste disposal.

In restoration technologies, the design of dry compatible mortar systems requires careful optimization of granulometric composition, binder–aggregate ratio and physicochemical interactions between components [10]. Shangina et al. [10] emphasized that restoration dry mixes should possess not only sufficient mechanical performance but also compatibility with the original masonry materials in terms of vapor permeability and salt transport behavior.

Despite numerous international studies devoted to restoration mortars, limited investigations have been conducted on restoration composites based on local Uzbek raw materials and industrial waste. In particular, insufficient attention has been paid to the development of restoration materials compatible with traditional “Hajja” mortars widely used in historical monuments of Khiva and other architectural complexes of Uzbekistan [1,2].

Therefore, the present study aims to develop heritage-compatible restoration mortars based on local loess-like raw materials and defecate-derived microcalcite for restoration and conservation of historical architectural monuments of Uzbekistan. The research focuses on physicochemical characterization, mineralogical compatibility and structural stability of experimentally developed restoration composites.

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2. MATERIALS AND METHODS

Experimental investigations were carried out using local mineral raw materials and industrial waste components compatible with traditional historical mortars of Uzbekistan. The main materials included loess-like soil, NaCl, $CaCO_3$, Fe_2O_3, quartz sand and defecate-derived microcalcite obtained from sugar industry waste. Historical "Hajja" mortar samples collected from the Khiva Ichan-Qala architectural complex were used as reference materials for comparative physicochemical analysis.

The loess-like soil collected from the Khorezm region mainly consisted of SiO_2, Al_2O_3, CaO and Fe_2O_3 phases. Defecate-derived microcalcite was used as a carbonate filler to improve structural density, pore filling and compatibility of the restoration mortars. The use of industrial waste also contributed to sustainable restoration technology development and waste valorization.

Prior to sample preparation, all raw materials were dried at 105 ± 5 °C for 24 h to remove residual moisture. The dried materials were milled using a laboratory ball mill until particle sizes of 0.1–0.5 mm were achieved. The obtained powders were sieved and homogenized in a laboratory mixer for 10–20 min to ensure uniform distribution of mineral components.

Experimental restoration compositions were prepared by varying the proportions of loess-like soil, NaCl, $CaCO_3$ and Fe_2O_3. The prepared mixtures were molded into cubic specimens with dimensions of 50 × 50 × 50 mm. The specimens were cured under laboratory conditions at a temperature of 20 ± 2 °C and relative humidity of approximately 95 % for 28 days to ensure stable carbonation and structural formation processes.

The chemical composition of raw materials and restoration composites was determined using X-ray fluorescence analysis (XRF). Mineralogical and structural investigations were carried out using Fourier transform infrared spectroscopy (FTIR), X-ray diffraction analysis (XRD), differential thermal analysis (DTA/DSC) and scanning electron microscopy (SEM). FTIR analysis was used to identify carbonate and silicate phases, while XRD investigations determined the crystalline composition of the developed restoration composites. DTA/DSC analysis was performed within the temperature range of 20–700 °C to evaluate thermal stability and phase transformations. SEM investigations were conducted to analyze pore structure, morphology of crystalline phases and structural densification.

The obtained physicochemical results were used to evaluate the structural compatibility of the developed restoration mortars with historical "Hajja" composites.

Table 1.

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Experimental compositions of restoration mortars

Composition	Loess (%)	NaCl (%)	CaCO3 (%)	Fe2O3 (%)
F-1	80	20	-	-
F-4	80	20	5	-
F-5	75	25	3	-
F-10	80	20	5	0.3

3. RESULTS AND DISCUSSION

The obtained experimental results demonstrated that the developed restoration composites exhibit high physicochemical and mineralogical compatibility with historical “Hajja” mortars. Comprehensive FTIR, XRD and DTA/DSC investigations confirmed the formation of stable carbonate–silicate structures within the developed restoration systems.

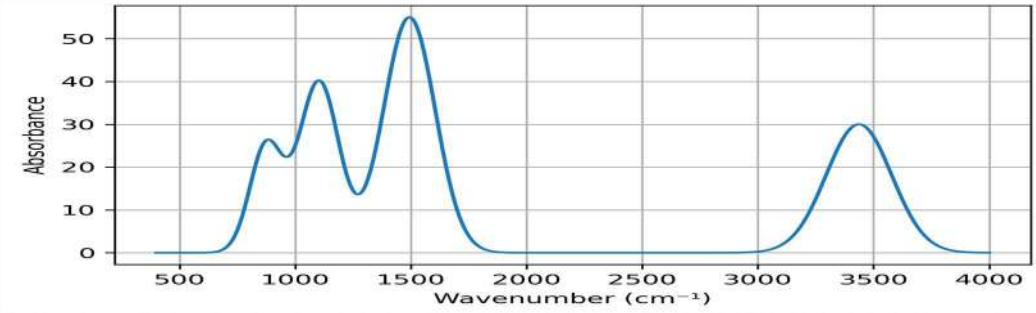


Figure 1. FTIR spectrum of historical “Hajja” mortar and restoration composite F-10

FTIR investigations confirmed the presence of carbonate, silicate and hydroxyl phases in the developed composites. The absorption bands observed near 3437 cm^{-1} correspond to hydroxyl groups and adsorbed moisture, while intensive peaks near 1494 cm^{-1} and 875 cm^{-1} indicate the presence of calcite phases. The absorption region near 1103 cm^{-1} corresponds to silicate structures associated with quartz and aluminosilicate minerals. The obtained spectra demonstrated strong similarity between the experimental compositions and historical “Hajja” mortars, indicating high mineralogical compatibility of the developed restoration materials.

The FTIR spectrum presented in Figure 1 demonstrates characteristic carbonate and silicate absorption bands. The high intensity of carbonate peaks confirms the significant role of $CaCO_3$ phases in structural stabilization and pore filling processes. The silicate absorption region indicates the presence of quartz and aluminosilicate components responsible for mechanical stability of the restoration composites.

XRD analysis identified NaCl, quartz and calcite as dominant crystalline phases within the developed restoration materials. The strongest diffraction peaks

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were observed at 2θ ≈ 31.7° and 45.4°, corresponding to NaCl phases, while diffraction peaks near 26.6° and 27.4° confirmed the presence of crystalline quartz. Additional low-intensity peaks indicated the presence of calcite and aluminosilicate phases.

The obtained XRD results demonstrated that the developed compositions reproduce the mineralogical structure of historical "Hajja" mortars with high accuracy. The presence of carbonate phases contributes to structural densification, while quartz particles improve mechanical stability and reduce shrinkage processes

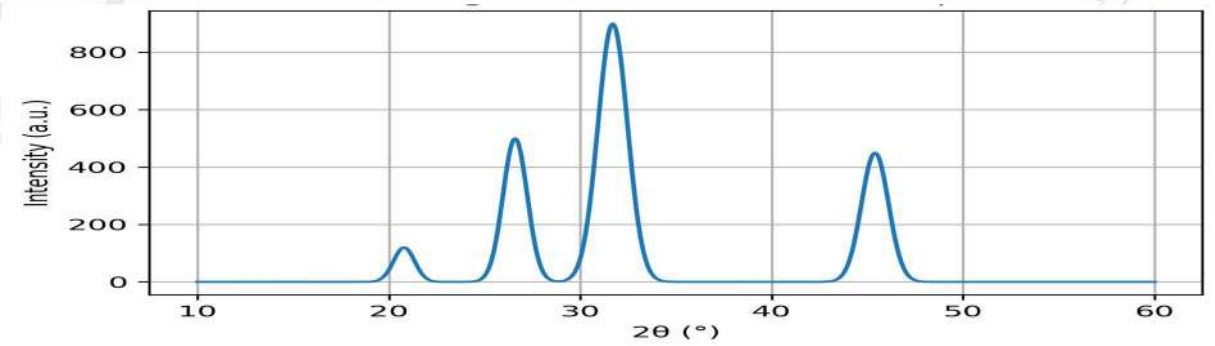


Figure 2. XRD diffractogram of restoration composite F-10

Table 2

Main crystalline phases identified by XRD analysis

2θ (°)	Phase	Relative intensity	Interpretation
20.8	Quartz/Calcite	Low	Minor mineral phase
26.6	Quartz	Medium	Silicate structure
27.4	Quartz	High	Main quartz phase
31.7	NaCl	Very high	Dominant crystalline phase
45.4	NaCl	High	Stable salt structure
50.5	Calcite	Medium	Carbonate phase

The XRD diffractogram presented in Figure 2 confirms the formation of stable crystalline phases within the restoration composite. The dominance of NaCl and quartz peaks indicates strong mineralogical similarity to historical mortars. The relatively uniform diffraction pattern also demonstrates homogeneous distribution of crystalline phases throughout the restoration matrix.

DTA/DSC investigations demonstrated stable thermal behavior of the developed restoration composites up to approximately 600 °C. Initial endothermic effects observed below 200 °C were associated with evaporation of physically bound moisture. Thermal effects within the range of 350–500 °C corresponded to dehydration and structural transformation of clay and aluminosilicate phases.

A significant endothermic effect observed near 640–680 °C was attributed to carbonate decomposition processes associated with calcite phases. The

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obtained results indicate good thermal stability of the restoration composites under moderate thermal conditions.

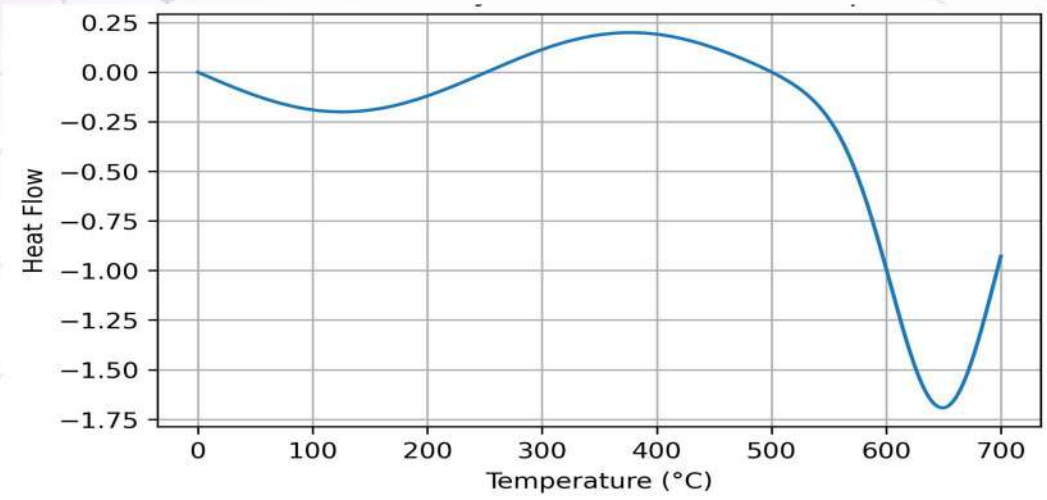


Figure 3. DTA/DSC analysis of restoration composite F-10

The thermal analysis curves presented in Figure 3 demonstrate gradual structural transformations without abrupt thermal destruction. The relatively stable thermal behavior confirms the suitability of the developed composites for restoration applications under environmental temperature fluctuations.

Table 3

Thermal effects identified by DTA/DSC analysis

Temperature range (°C)	**Thermal effect**	**Interpretation**
100–200	Endothermic	Moisture evaporation
350–500	Endothermic	Dehydration of mineral phases
560–600	Weak thermal effect	Structural transformation
640–680	Intensive endothermic	Carbonate decomposition

Comparative analysis of the experimental compositions demonstrated that the addition of $CaCO_3$ significantly improves structural density and reduces porosity. Compositions containing optimized NaCl concentrations exhibited stable crystallization behavior and improved structural integrity.

Among the investigated compositions, sample F-10 demonstrated the highest structural compatibility with historical “Hajja” mortars. The developed structure was characterized by dense packing of mineral particles, low pore volume and improved adhesion between crystalline phases.

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Table 4

Comparative characteristics of experimental composites

Composition	Structural density	Porosity	Compatibility
F-1	Medium	Medium	Satisfactory
F-4	High	Low	Good
F-5	High	Low	Good
F-10	Very high	Very low	Maximum

The obtained results indicate that the combined use of loess-like soil, carbonate fillers and controlled NaCl content allows the development of restoration composites highly compatible with historical architectural mortars. The use of defecate-derived microcalcite additionally contributes to sustainable waste utilization and environmental protection.

4. CONCLUSIONS.

The conducted investigations confirmed the possibility of producing heritage-compatible restoration mortars using local raw materials and defecate-derived microcalcite. FTIR and XRD analyses demonstrated high mineralogical similarity between the developed composites and traditional historical “Hajja” mortars. DTA/DSC investigations confirmed stable thermal behavior of the restoration materials up to 600 °C.

Comparative analysis showed that the addition of $CaCO_3$ and controlled NaCl content improved structural density and reduced porosity of the restoration composites. Among the investigated compositions, F-10 exhibited the highest compatibility with historical mortars due to its dense microstructure, low porosity and improved adhesion behavior.

The obtained results indicate that local mineral resources and industrial waste can be effectively utilized for sustainable restoration and conservation of historical architectural monuments of Uzbekistan.

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