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CIRCULAR VALORIZATION OF SPENT INDUSTRIAL ZEOLITE ADSORBENTS INTO DIFFERENTIATED SOIL-IMPROVING PRODUCTS: REGENERATION, CHARACTERIZATION AND PRELIMINARY PRODUCT-SPECIFIC ASSESSMENT

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ABSTRACT

Spent industrial zeolite adsorbents from gas drying, hydrocarbon purification and molecular-sieve separation units represent a mineral waste stream with potential value for sustainable soil management. This study develops a product-specific circular route for converting CaA-, NaA- and KA-type spent zeolites into three soil-improving products: ZeoMeliorant-CaA for saline and compacted soils, ZeoModifier-NaA/KA for nutrient retention in cultivated and greenhouse soils, and ZeoSubstrate-15 for peat-based growing media. The proposed route combines mechanical preparation, thermal regeneration at 530–550 °C, optional microwave activation, mild citric-acid modification, washing, drying and fractionation. The manuscript also establishes a soil-testing matrix and safety-control criteria for agricultural use. Preliminary model-based assessment indicates that properly regenerated zeolite products may improve water-holding capacity, cation-exchange performance and biological response while reducing salinity-related stress when the dose and target medium are correctly selected. FTIR and XRD characterization are used to verify removal of organic residues, surface modification and preservation of the A-type/LTA aluminosilicate framework. The approach links industrial adsorbent waste management with soil melioration, nutrient retention and substrate improvement, but final agronomic validation requires measured XRF/ICP, FTIR, XRD, pH, EC, CEC, residual hydrocarbon, heavy-metal, germination and pot-trial data.

Keywords: spent zeolite, molecular sieve, soil meliorant, soil amendment, cation exchange capacity, salinity, peat substrate, circular economy, FTIR, XRD

ANNOTATSIYA

Gazni quritish, uglevodorodlarni tozalash va molekulyar elaklarda ajratish qurilmalaridan chiqqan ishlangan sanoat seolit adsorbentlari tuproqni barqaror boshqarish uchun potentsial ahamiyatga ega bo'lgan mineral chiqindilar oqimini ifodalaydi. Ushbu tadqiqot CaA-, NaA- va KA- tipli ishlangan seolitlarni tuproqni yaxshilovchi uchta mahsulotga aylantirish uchun o'ziga xos aylanma marshrutni ishlab chiqadi: sho'r va zichlashgan tuproqlar uchun ZeoMeliorant-CaA, ekin maydonlari va issiqxona tuproqlarida ozuqa moddalarini saqlash uchun ZeoModifier-NaA/KA va torf asosidagi o'stirish muhitlari uchun ZeoSubstrate-15. Taklif etilayotgan usul mexanik tayyorlash, 530–550 °C da termik regeneratsiya, ixtiyoriy mikroto'lqinli faollashtirish, limon kislotasi bilan yumshoq modifikatsiya, yuvish, quritish va fraksiyalashni o'z ichiga oladi. Maqolada, shuningdek, qishloq xo'jaligida foydalanish uchun tuproqni sinash matritsasi va xavfsizlikni nazorat qilish mezonlari belgilanadi. Dastlabki modelga asoslangan baholashlar shuni ko'rsatadiki, to'g'ri regeneratsiya qilingan seolit mahsulotlari doza va maqsadli muhit to'g'ri tanlanganda suvni ushlab turish qobiliyatini, kation almashinish faoliyatini va biologik reaksiyani yaxshilashi hamda sho'rlanish bilan bog'liq stressni kamaytirishi mumkin. FTIR va XRD tahlillari organik qoldiqlarning olib tashlanganligini, sirt o'zgarishini va A-tipi/LTA alumosilikat strukturasining saqlanib qolganligini tekshirish uchun ishlatiladi. Ushbu yondashuv ishlangan sanoat adsorbent chiqindilarini boshqarishni tuproq melioratsiyasi, ozuqa moddalarini saqlash va substratni yaxshilash bilan bog'laydi, ammo yakuniy agronomik tasdiqlash uchun o'lchangan XRF/ICP, FTIR, XRD, pH, EC, CEC, qoldiq uglevodorodlar, og'ir metallar, unib chiqish va tuval tajriba ma'lumotlari talab qilinadi.

Kalit so'zlar: ishlangan seolit; molekulyar elak; tuproq melioranti; tuproqni yaxshilovchi; kation almashinish sig'imi; sho'rlanish; torf substrati; aylanma iqtisodiyot; FTIR; XRD

АННОТАЦИЯ

Отработанные промышленные цеолитные адсорбенты из установок осушки газа, очистки углеводородов и молекулярно-ситового разделения представляют собой поток минеральных отходов, имеющий потенциальную ценность для устойчивого управления почвами. В данном исследовании разрабатывается специфичный циркулярный маршрут для переработки отработанных цеолитов типов CaA, NaA и KA в три продукта для улучшения почвы: ZeoMeliorant-CaA для засоленных и уплотненных почв, ZeoModifier-NaA/KA для удержания питательных веществ в культивируемых и тепличных почвах и ZeoSubstrate-15 для торфяных питательных сред. Предлагаемый маршрут включает механическую подготовку, термическую регенерацию при 530–550 °C, опциональную микроволновую активацию, мягкую модификацию лимонной кислотой, промывку, сушку и фракционирование. В статье также устанавливаются матрица тестирования почвы и критерии контроля безопасности для сельскохозяйственного использования. Предварительная оценка на основе моделей показывает, что правильно регенерированные цеолитные продукты могут улучшить влагоемкость, катионообменную способность и биологический отклик, одновременно снижая стресс, связанный с засолением, при правильном выборе дозы и целевой среды. Характеристики FTIR и XRD используются для проверки удаления органических остатков, модификации поверхности и сохранения алюмосиликатного каркаса типа A/LTA. Подход связывает управление отходами промышленных адсорбентов с мелиорацией почв, удержанием питательных веществ и улучшением субстрата, однако для окончательной агрономической валидации требуются измеренные данные XRF/ICP, FTIR, XRD, pH, EC, CEC, остаточных углеводородов, тяжелых металлов, всхожести и вегетационных опытов.

Ключевые слова: отработанный цеолит; молекулярное сито; почвенный мелиорант; улучшитель почвы; емкость катионного обмена; засоленность; торфяной субстрат; циркулярная экономика; FTIR; XRD

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1. Introduction

Zeolites are crystalline aluminosilicates with permanent microporosity, exchangeable cations and a strong affinity toward water, ammonium and other polar species. These properties explain their industrial use as molecular sieves as well as their growing role in soil conditioning, nutrient management and environmental remediation [1–5].

In agricultural systems, zeolitic materials can improve water retention, reduce nutrient losses, buffer ammonium and potassium in the root zone, and help stabilize selected contaminants. Their performance, however, depends on mineral type, particle size, exchangeable cation form, application rate and the chemical condition of the soil [6–11].

Most published studies focus on natural clinoptilolite or newly synthesized zeolites. In contrast, the direct conversion of spent industrial CaA, NaA and KA molecular sieves into safe soil-improving products remains less developed. This gap is important because spent adsorbents are usually treated as waste even though their aluminosilicate framework may remain suitable for secondary use after proper regeneration and safety control [12–14].

The key principle of the present work is product differentiation. A Ca-rich zeolite fraction is more suitable for meliorative use in saline or compacted soils, where calcium exchange and moisture retention may support structure improvement. NaA/KA fractions are better positioned as nutrient-retention modifiers in non-saline soils, where excessive sodium input is not a limiting factor. Fine regenerated zeolite blends can be used in peat-based substrates at controlled rates. On this basis, the study proposes a circular technological route and a product-specific testing framework for spent zeolite valorization.

Circular valorization scheme for converting spent industrial CaA/NaA/KA zeolite adsorbents into differentiated soil-improving products. The route includes mechanical sorting, thermal regeneration, mild citric-acid modification, washing, drying, fractionation and product routing into ZeoMeliorant-CaA, ZeoModifier-NaA/KA and ZeoSubstrate-15.

The proposed concept is based on a stepwise conversion of spent industrial zeolite adsorbents into three product-oriented soil amendments. As shown in Figure 1, the process combines mechanical preparation, regeneration, mild surface modification and fractionation. This approach allows CaA-, NaA/KA- and fine zeolite-rich fractions to be directed toward different agronomic uses rather than being applied as a single undifferentiated amendment.

2. Materials and Methods

Raw material and product concept

The raw material is represented by spent industrial molecular-sieve adsorbents containing CaA, NaA and KA fractions after service in adsorption, drying or gas-treatment units. Before agricultural routing, each batch should be screened for residual hydrocarbons, sulfur-containing compounds, soluble salts, toxic metals, pH and particle-size distribution. Only material passing the safety criteria should be used for soil or substrate applications.

Technological conversion route

The proposed process includes mechanical preparation, thermal regeneration, optional microwave activation, mild organic-acid modification, washing, drying and fractionation. Sorting and sieving remove dust and non-target fractions. Thermal regeneration at 530–550 °C is used to remove volatile and organic residues without collapsing the aluminosilicate framework. Microwave activation may be applied as an optional intensification step. Citric-acid treatment is selected as a mild modification route because it can clean and functionalize the surface without introducing aggressive inorganic residues.

Figure 1. Proposed circular technology for converting spent zeolite adsorbents into three soil-improving products.

Experimental design and analytical control

Each product should be tested against a no-zeolite control with at least three independent replicates. The minimum dataset should include soil pH, electrical conductivity (EC), water-holding capacity (WHC), bulk density, cation exchange capacity (CEC), available nitrogen, phosphorus and potassium, germination index, root development and biomass response. Product safety must be assessed by determining residual petroleum hydrocarbons and toxic metals in both the amended material and treated soil [19–23].

3. Preliminary Product-Specific Assessment

The values in this section are presented as a model-based assessment for planning the experimental program and selecting initial dose ranges. The same layout can be retained after replacing the values with measured mean ± standard deviation, statistical grouping and exact p-values.

Product performance matrix

Dose-response behavior

Dose-response curves are more informative than a single treatment comparison because they show whether the effect increases linearly, reaches a plateau or becomes risky at higher rates. For the proposed products, water retention should be tested across a practical dose range, while salinity response should be validated in incubation, column or pot experiments using the CaA-rich meliorant [2,6–8,19].

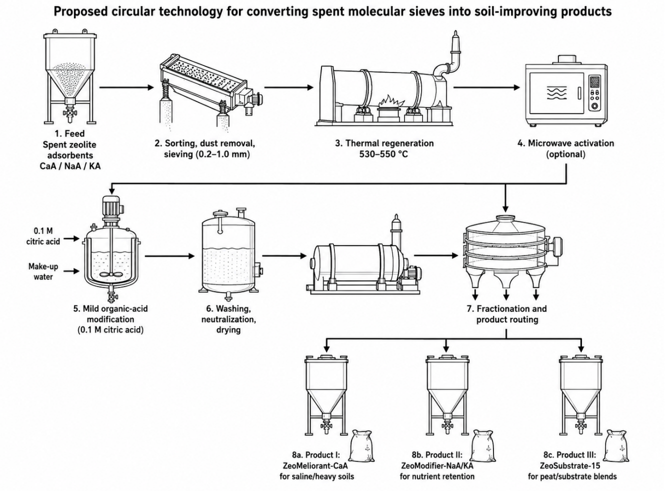

Figure 2. Model-based dose-response curves for water retention of the three product types.

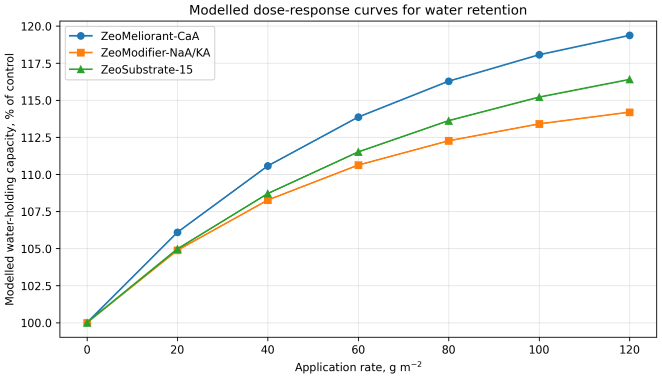

Figure 3. Model-based response of electrical conductivity and exchangeable sodium to ZeoMeliorant-CaA dose in saline soil.

FTIR and XRD verification

A soil-use technology based on spent adsorbents must demonstrate that regeneration removes undesirable residues and that the zeolitic framework remains sufficiently preserved. FTIR is suitable for monitoring surface hydroxyl groups, adsorbed water, aluminosilicate bands and citrate-related functional groups. XRD verifies whether the A-type/LTA crystalline framework survives thermal regeneration and mild acid modification [15–18,24,25].

The main reflections observed in the XRD pattern are assigned to A-type/LTA zeolite framework positions. The peak around 26.9° is treated cautiously because it may overlap with a quartz-like siliceous impurity; this assignment should be confirmed by complementary XRF, Rietveld refinement or reference-pattern comparison.

Table 1. Differentiated product concept for spent zeolite valorization.

Product	Dominant zeolite form	Target function	Preliminary dose	Priority environment	Main restriction
ZeoMeliorant-CaA	CaA-rich fraction	Water retention, structure improvement and partial salinity mitigation	60–120 g m^{-2} or 3–7.5 t ha^{-1}	Saline, compacted, heavy or sandy soils	Use only after residue and heavy-metal screening
ZeoModifier-NaA/KA	NaA/KA fraction	Nutrient retention and cation-exchange buffer for N and K fertilizers	30–80 g m^{-2} or 2–5 t ha^{-1}	Cultivated loam and greenhouse soil	Avoid high doses in saline or sodic soils
ZeoSubstrate-15	Regenerated fine zeolite blend	Peat/substrate additive for moisture and nutrient-release stabilization	8–10 g dm^{-3} or 15 vol.%	Seedling and greenhouse substrate	Check pH and EC before seedling use

Table 2. Analytical control plan and acceptance criteria for pilot product screening.

Indicator	Unit	Minimum method	Acceptance criterion for pilot product
pH	pH units	Potentiometric soil/water or substrate/water extract	Within target range of the selected soil or substrate
Electrical conductivity	dS m^{-1}	Conductometric extract	No unsafe increase above soil/substrate class limit
Water-holding capacity	% of dry soil/substrate	Gravimetric saturation-drainage method	At least 10% improvement for meliorant/substrate use
Cation exchange capacity	cmol(+) kg^{-1}	Ammonium acetate or equivalent method	At least 10% improvement over control
Petroleum hydrocarbons	mg kg^{-1}	IR or GC-based method	Below applicable agricultural safety threshold
Toxic metals	mg kg^{-1}	ICP-OES, ICP-MS or AAS	Below soil-amendment safety limits
Germination index	%	Seed germination bioassay	At least 80% and not lower than control
Biomass response	% of control	Pot trial, dry mass	Positive trend, preferably ≥8–10% increase

Table 3. Preliminary model-based performance matrix for selecting experimental doses.

Product / medium	Dose	pH: control → treated	EC: control → treated	WHC, % of control	CEC, % of control	Biological response, % of control	Recommended criterion
ZeoMeliorant-CaA / saline soil	100 g m^{-2}	8.4 ± 0.1 → 8.0 ± 0.1	3.8 ± 0.2 → 2.9 ± 0.2	118 ± 3	112 ± 4	112 ± 5	EC reduction ≥15%; no phytotoxicity
ZeoMeliorant-CaA / heavy loam	80 g m^{-2}	7.8 ± 0.1 → 7.6 ± 0.1	1.6 ± 0.1 → 1.5 ± 0.1	115 ± 3	110 ± 3	111 ± 4	Positive root response or lower bulk density
ZeoMeliorant-CaA / sandy soil	100 g m^{-2}	7.4 ± 0.1 → 7.5 ± 0.1	1.1 ± 0.1 → 1.2 ± 0.1	122 ± 4	116 ± 4	116 ± 5	WHC increase ≥15%
ZeoModifier-NaA/KA / cultivated loam	60 g m^{-2}	7.2 ± 0.1 → 7.4 ± 0.1	1.3 ± 0.1 → 1.4 ± 0.1	112 ± 3	122 ± 5	110 ± 4	CEC and N/K retention ≥10%
ZeoModifier-NaA/KA / greenhouse soil	50 g m^{-2}	6.8 ± 0.1 → 7.0 ± 0.1	1.8 ± 0.1 → 1.9 ± 0.1	111 ± 3	118 ± 4	113 ± 5	No EC accumulation; fertilizer-saving potential
ZeoModifier-NaA/KA / saline-prone soil	30 g m^{-2}	8.1 ± 0.1 → 8.2 ± 0.1	2.7 ± 0.2 → 2.8 ± 0.2	106 ± 2	110 ± 3	105 ± 3	Low-dose use only; Na risk controlled
ZeoSubstrate-15 / peat substrate	15 vol.%	5.7 ± 0.1 → 6.3 ± 0.1	0.8 ± 0.1 → 0.9 ± 0.1	118 ± 4	128 ± 5	117 ± 4	GI ≥90%; pH 5.8–6.8
ZeoSubstrate-15 / seedling mix	8–10 g dm^{-3}	6.2 ± 0.1 → 6.5 ± 0.1	1.0 ± 0.1 → 1.1 ± 0.1	114 ± 3	121 ± 4	115 ± 5	Uniform seedling growth; no chlorosis
ZeoSubstrate-15 / local planting zone	50–80 g m^{-2}	7.0 ± 0.1 → 7.1 ± 0.1	1.2 ± 0.1 → 1.2 ± 0.1	110 ± 3	114 ± 3	109 ± 4	Positive local root-zone effect

Table 4. XRD peak assignment of A-type/LTA zeolite phase.

No.	2θ, degree	d-spacing, Å	Intensity class	Assigned phase	Structural interpretation	Relevance to soil-improving product
1	7.2	12.27	VS	Zeolite A-type / LTA	Main low-angle reflection of crystalline LTA framework	Confirms preservation of microporous aluminosilicate structure
2	10.1	8.75	S	Zeolite A-type / LTA	Characteristic reflection of ordered aluminosilicate lattice	Supports ion-exchange potential
3	12.4	7.13	S	Zeolite A-type / LTA	Confirms crystalline zeolite A phase	Indicates phase stability after regeneration
4	16.0	5.54	M	Zeolite A-type / LTA	Secondary framework reflection	Shows that thermal treatment did not destroy the structure
5	21.7	4.09	M–S	Zeolite A-type / LTA	Retained zeolitic lattice ordering	Related to retained crystalline ordering
6	24.0	3.71	M	Zeolite A-type / LTA	Aluminosilicate framework reflection	Supports nutrient-retention function
7	25.8	3.45	M	Zeolite A-type / LTA	Framework-related reflection	Indicates retained mineral skeleton
8	26.9	3.31	W–M	Zeolite A-type / possible silica overlap	Possible overlap with quartz-like siliceous impurity	Should be controlled by XRF or phase analysis
9	30.0	2.98	M–S	Zeolite A-type / LTA	Characteristic zeolitic reflection	Confirms structural integrity after modification
10	30.8	2.90	M	Zeolite A-type / LTA	Additional LTA-related reflection	Supports stability of the regenerated adsorbent
11	31.6	2.83	M	Zeolite A-type / LTA	Framework ordering after regeneration	Confirms ordered crystalline phase
12	34.0	2.64	W–M	Zeolite A-type / LTA	Secondary reflection of preserved crystalline phase	Indicates preserved zeolite phase

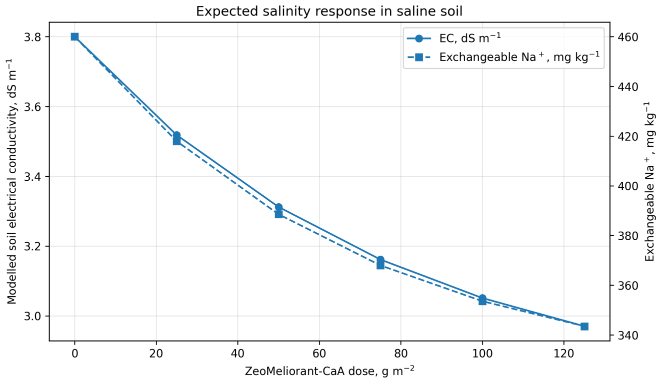

Figure 4. Representative FTIR spectra of spent zeolite adsorbent (SS), thermally regenerated zeolite (RZ), and citric-acid-modified spent zeolite (mSS). Bands at 3430–3435 and 1630–1635 cm^{-1} correspond to surface hydroxyl groups and adsorbed water; bands in the 1200–700 cm^{-1} region are mainly attributed to aluminosilicate framework vibrations. Additional bands in mSS near 2930, 1270, 1130 and 1025 cm^{-1} indicate surface modification by citrate-containing groups.

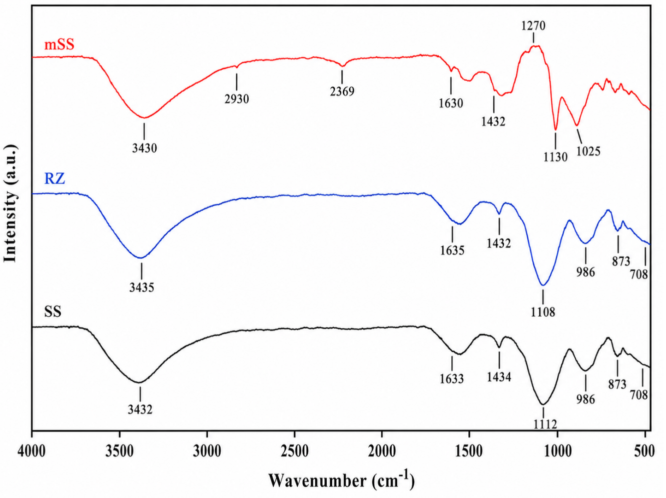

Figure 5. XRD fingerprint of the regenerated/modified spent zeolite adsorbent showing characteristic reflections of the zeolite A-type/LTA crystalline phase.

4. Discussion

The proposed approach is based on the idea that spent zeolite adsorbents should not be converted into a single undifferentiated soil additive. Their use should depend on cation form, expected soil response and safety status. A CaA-rich product is preferable for meliorative application in saline or compacted soils because calcium exchange and water retention can support aggregation and reduce sodium-related stress. NaA/KA fractions are better suited to nutrient retention in cultivated and greenhouse soils, where soluble-salt accumulation can be controlled. A fine regenerated blend is most appropriate for peat-based substrates, where dose, pH and EC can be adjusted before plant use [1–11].

The model-based performance matrix suggests that the greatest benefit should be expected where the zeolite function matches the soil limitation. In sandy or peat-rich media, the main benefit is water retention. In cultivated loam, the main benefit is cation exchange and nutrient retention. In saline soil, the CaA-rich product is more defensible than Na-rich products because sodium addition may worsen sodicity if the dose is not controlled.

The characterization strategy is essential for scientific credibility. FTIR should show reduced organic residue signals after regeneration and the presence of framework bands after modification. XRD should confirm that the A-type/LTA structure remains crystalline enough to support adsorption and ion-exchange behavior. These analyses do not replace agronomic testing, but they justify why the regenerated material can function as a soil-improving product.

Safety validation is the critical boundary condition for this technology. Industrial molecular sieves may contain hydrocarbons, sulfur compounds, process contaminants or trace metals depending on service history. Therefore, agricultural reuse is acceptable only after regeneration and analytical proof that the final product does not introduce phytotoxic or hazardous residues. This requirement should be presented as a central part of the technology rather than a supplementary check [12–14,20–23].

Compared with conventional zeolite-amendment studies, the novelty of the present approach is the circular conversion of spent industrial

Table 5. Experimental evidence required for converting the framework into a final research article.

Manuscript element	Current role in this manuscript	Required final evidence
Technology scheme	Conceptual process route	Actual process parameters, mass yield and material loss
Water-retention curves	Model-based planning data	Measured WHC at 4–6 doses with n ≥ 3
Salinity response	Model-based trend	EC, exchangeable Na, SAR/ESP after incubation or column testing
FTIR spectrum	Representative characterization format	Measured spectra of spent, regenerated and modified samples
XRD pattern	Representative phase-verification format	Measured diffractograms and crystallinity comparison
Results tables	Preliminary mean ± SD format	Measured values, p-values and statistical grouping
Safety control	Acceptance criteria	ICP/AAS metal data and residual hydrocarbon analysis
Agronomic response	Predicted response	Germination, root/shoot ratio, biomass and leachate data

molecular sieves into product-specific amendments with separate target environments and dose criteria. The strongest final article will combine material characterization, soil incubation, germination bioassay, pot experiments and leachate nutrient analysis.

5. Final Experimental Validation Plan

Table 5 outlines the required experimental evidence for validating the proposed framework and converting it into a final research article.

6. Conclusions

Spent CaA, NaA and KA zeolite adsorbents can be considered promising secondary mineral resources for soil-improving products when regeneration, safety screening and product-specific routing are applied. The proposed technology differentiates three products: ZeoMeliorant-CaA for saline and compacted soils, ZeoModifier-NaA/KA for nutrient retention in cultivated and greenhouse soils, and ZeoSubstrate-15 for peat-based growing media. This differentiation is important because the same zeolite material may be beneficial in one soil environment and risky or inefficient in another.

The preliminary assessment indicates that the most defensible product claims are improved water retention, increased cation-exchange buffering, better nutrient retention and reduced salinity stress under controlled dose conditions. FTIR and XRD provide the material-level evidence needed to support these claims by verifying surface cleaning, modification and framework preservation. Final journal submission should be based on measured analytical and agronomic data, including XRF/ICP, FTIR, XRD, pH, EC, CEC, residual hydrocarbons, toxic metals, germination index and pot-trial performance.

Overall, the concept provides a practical circular-economy pathway for converting industrial adsorbent waste into differentiated soil amendments. Its scientific strength will depend on rigorous safety validation and statistically supported soil and plant-response data.

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