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Effect of wood vinegar on the release of calcium, magnesium, and phosphorus from calcareous soils in different land uses

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Abstract: The release of essential nutrients from soil minerals for plant growth in calcareous soils, facilitated by organic extractants, is critical in semi-arid areas, particularly for elements affected by high soil pH. This study aims to investigate the release of calcium (Ca), magnesium (Mg), and phosphorus (P) through the application of wood vinegar extract in surface calcareous soils in Borojerd City, Lorestan Province, Iran. The experiment was conducted using a completely randomized design with three replications. The treatments included soils from three different land uses: vineyard, wheat field, and rangeland, each treated with 1.00% wood vinegar solution. Cumulative measurements of the specified elements were recorded over 10 consecutive 0.5 h intervals. The release data were analyzed using four various kinetic models (Elovich equation, parabolic diffusion law, power function equation, and zero-order kinetics). The highest concentrations recorded were for Ca (39,500.00 mg/kg), Mg (5880.00 mg/kg), and P (5.00 mg/kg) in grape cultivation. The findings revealed a significant difference in Ca release between grape cultivation and rangeland ($P<0.01$), while the Mg release showed a significant difference between both grape cultivation and rangeland and wheat cultivation ($P<0.01$). Additionally, the cumulative release of P showed significant differences between grape cultivation and both wheat and rangeland ($P<0.01$). The results indicated that the zero-order kinetics provided the best fit for the data ($R^2=0.99$). The maximum initial release amount was observed in grape cultivation when applying the zero-order kinetics, while the highest release rate was achieved using the parabolic diffusion law across three applications. Wood vinegar had the capacity to degrade various clay minerals, including vermiculite, smectite, palygorskite, and, to some extent, illite, resulting in the release of associated elements. Consequently, it can be concluded that wood vinegar can be effectively utilized in grape cultivation as an agent for reducing soil acidity, thereby enhancing the availability of soil nutrients and decreasing reliance on chemical fertilizers.

Keywords: X-ray diffraction; kinetics analysis; vineyard; wheat field; rangeland; vermiculite

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1 Introduction

Minerals present in the soil act as both a source and a reservoir for essential nutrients required by plants. Permanently charged phyllosilicates, such as vermiculite, mica, and smectite, contain exchange sites that retain essential nutrients in cationic form on their inner and outer surfaces (Sollins et al., 1988). Among these essential elements, calcium (Ca), the seventh most abundant element in the Earth's crust, plays a critical role in maintaining the stability and structural integrity

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of cell wall membranes. Magnesium (Mg), the eighth most abundant element in the Earth's crust, is classified as an essential nutrient for plants and is vital for their physiological processes. The bioavailability of Mg, along with other essential elements released from clay minerals, is influenced by several factors, including soil acidity, particle size, and varying weather conditions. In acidic soils, Mg is predominantly present as Mg ions, which are readily accessible to plants. Conversely, in calcareous soils, the interaction between Mg ions and carbonate ions leads to the formation of insoluble magnesium carbonate ($MgCO_3$), rendering Mg unavailable to plants (Hailes et al., 1997). Dash et al. (2023) found that magnesium sulfate ($MgSO_4$) released a greater quantity of Mg ions compared with $MgCO_3$ and dolomite. The majority of Mg in soil, estimated to be between 90.00% and 98.00%, is incorporated within the crystalline structure of mineral materials, rendering it unavailable for direct uptake by plants (Gransee and Führs, 2013). Phosphorus (P) ranks as the eleventh most abundant element on the Earth. Due to its high reactivity in natural environments, P is not readily available in its elemental form. Its availability is further diminished by fixation processes in the soil, which occur through reactions with soluble Ca in calcareous soils and with iron and aluminum in acidic soils. During the growing season, only a small fraction of the total P present in the soil is accessible to plants, and the effectiveness of chemical P fertilizers diminishes more rapidly than that of other nutrient elements. Additionally, sediments and soils contain substantial quantities of organic, mineral, and microbial P, indicating that P dynamics are influenced by a complex interplay of chemical and biological processes within the soil (Srinath Reddy et al., 2005).

Both organic and inorganic acids significantly influence the release of elements from soil minerals. Research has shown that organic acids produced by microorganisms and plants enhance the dissolution of minerals in weathered environments (Bartlett et al., 2018). The application of acids, in conjunction with chemical fertilizers, improves the absorption capacity of specific nutrients, ultimately leading to increased agricultural yields. Ström et al. (2005) found that applying oxalic and citric acid solutions with pH values below 3.50 enhanced the release of Ca and Mg from calcareous soil. Notably, citric acid was found to release nine times more Ca and twice as much Mg compared with oxalic acid. Jenagh et al. (2015) demonstrated that the release of specific elements from sepiolite and palygorskite in saline soils is significantly influenced by the presence of two types of organic acids: citric acid and oxalic acid, both at a concentration of 10 mM. Their findings indicated that oxalic acid is an effective extractant for iron (Fe), while citric acid is more suitable for extracting Mg from bentonite minerals. Mohammad Jafari et al. (2015) conducted a study examining the impact of two organic acids, oxalic acid, and citric acid, on the rate of Mg release from sepiolite mineral. The results indicated that the amount of Mg released from samples treated with citric acid is greater than that treated with oxalic acid. They observed that the rate of Mg release increases as the pH of environment decreases and as the particle size of the sepiolite mineral decreases. Lü et al. (2015) demonstrated that varying concentrations of organic acids significantly influenced P release. When the concentration was reduced to 1 mM, both the total and the maximum amounts of soil P released exhibited a significant decline, following this order of effectiveness: citric acid<malic acid<oxalic acid<acetic acid. Liu et al. (2016) revealed that Ca release induced by organic acids, specifically citric acid and oxalic acid, was enhanced in calcareous soils. Chahardoli (2022) demonstrated that among the extraction methods utilizing calcium chloride ($CaCl_2$), citric acid, oxalic acid, and hydrochloric acid, citric acid was the most effective extractant for releasing Mg and Ca from vermiculite clay soil.

Numerous studies have investigated the use of organic and inorganic extractants for extracting various elements from clay minerals. The results indicated a more favorable effect of organic extractants compared with inorganic ones. It is assumed that wood vinegar, as an organic extractant, is more effective than mineral extractants. However, there is limited information available regarding the use of wood vinegar for the extraction of Mg, Ca, and P from soil minerals. Wood vinegar, which contains a high concentration of acetic acid, promotes the release of Mg, Ca, and P from the soil by lowering soil pH. Recently, wood vinegar has garnered attention due to its innovative properties and low production costs. In addition to promoting vegetative and reproductive growth in various plant species, this substance has also proven effective in controlling a range of diseases and pests (Chen et al., 2020; Zhu et al., 2021). The application of wood vinegar has been shown to enhance various chemical properties of infertile soils in tropical regions, including increased acidity (Li et al., 2015; Najafi-Ghiri et al., 2024), total nitrogen content (Lu et al., 2019), available P (Yatagai et al., 2002), P exchange capacity, exchangeable cations, and saturation levels (Lu et al., 2019). Heshmati et al. (2023) demonstrated that wood vinegar significantly enhanced the release of non-exchangeable potassium (K) in soils used for grape cultivation, particularly when compared with citric and oxalic acids. Najafi-Ghiri et al. (2024) investigated the impact of wood vinegar on K release and found that adding wood vinegar to the soil resulted in a significantly greater release of K compared with the use of $CaCl_2$ extract. Idowu et al. (2023) conducted a study examining the effects of wood vinegar and biochar on potato cultivation. The findings indicated that the combination of these two treatments enhanced potato yield. Implementing innovative methodologies aimed at reducing the pH of rhizosphere, facilitating the release of specific elements from clay minerals, and improving the absorption of Mg and P by plants is essential. The reducing of soil pH provides valuable insights into the concentrations of Mg and P sources present in the soil, thereby contributing to a more comprehensive understanding of the chemical and biological processes associated with mineral substances, particularly Mg, in both soil and plant systems. Given the beneficial effects of wood vinegar on enhancing soil organic carbon (Wang et al., 2018), which is attributed the presence of various component organic substances, including organic acids, ketones, aldehydes, alcohols, and benzene and its derivatives (Lu et al., 2019), as well as the ability to improve nutrient availability stimulating soil microbial activity (Chen et al., 2020).

The arid and semi-arid soils of Iran are characterized by high lime content, which significantly limits the availability of essential elements such as Mg and P. Wood vinegar, which contains a substantial proportion of acetic acid, offers a cost-effective organic acid that may provide beneficial effects in agricultural practices on calcareous soils in Iran. Therefore, the objective of this study is to examine the release of Ca, Mg, and P through the application of industrial organic acid (wood vinegar) across three different usage systems (vineyard, wheat field, and rangeland) to compare the beneficial effects of wood vinegar among these three land uses and to evaluate its optimal application for land management.

2 Materials and methods

2.1 Sampling and measurement of physical-chemical characteristics

We collected soil samples from the surface horizon (0–20 cm). The sampling area is located in the alluvial plain of Borojerd City in Lorestan Province, Iran, which is situated between 34°01′–34°40′N and 48°22′–49°10′E, under a semi-arid climate characterized by cold winters and mild and dry summers. We collected three soil samples from each crop land type. Then, we evaluated various physical and chemical properties of the soil samples, including soil acidity (Thomas, 1996), electrical conductivity (EC) (Rhoades, 1996), cation exchange capacity (CEC) (Chapman, 1965), calcium carbonate equivalent (CCE) (Loeppert and Suarez, 1996), organic carbon (Nelson and Sommers, 1996), and soil texture (Gee and Bauder, 1986). Furthermore, we measured the concentrations of soluble Ca and Mg in the soil using the titration method with ethylenediaminetetraacetic acid (EDTA) (Rowell, 2014) and assessed the available P according to the Olsen et al. (1954).

2.2 Wood vinegar preparation and analysis

The wood vinegar used in this study was sourced from a charcoal production facility located in the Darab Region of southern Fars Province, Iran, the Fifth Quarter Company. This facility, which includes an electric furnace as well as purification and filtration devices, produces biochar from fruit tree wood at a temperature of 400°C under conditions of limited oxygen. We assessed various chemical properties of the wood vinegar sample using established standard methodologies. Fourier-transform infrared spectroscopy (FTIR) was employed, specifically utilizing the potassium bromide (KBr) pellet technique with a Shimadzu DR-8001 instrument (Shimadzu Company, Kyoto, Japan), to analyze and identify the functional groups present in the wood vinegar sample. The wood vinegar has a pH of 3.54, contains 10.11% organic carbon, and exhibits an EC of 5.15 dS/m (Table 1). The prominent absorption bands observed in the spectral regions of 2800–3800, 1600–1700, and 1290 /cm are primarily attributed to the stretching vibrations of C–H and O–H, C=O, and C–OH functional groups, respectively (Najafi-Ghiri et al., 2024). These spectral features likely indicated the presence of acetic acid and water, as illustrated in Figure 1.

Table 1 Property of the wood vinegar used in this study

Substance	pH	EC (dS/m)	Content (%)		Concentration (mg/kg)					
			OC	TN	P	K	Fe	Mn	Zn	Cu
Wood vinegar	3.54	5.15	10.11	0.05	Trace	0.01	40.42	Trace	5.51	2.81

Note: EC, electrical conductivity; OC, organic carbon; TN, total nitrogen; P, phosphorus; K, potassium; Fe, ferrum; Mn, manganuse; Zn, zinc; Cu, copper. Trace means the substance is undetectable with Inductively Coupled Plasma (ICP) device.

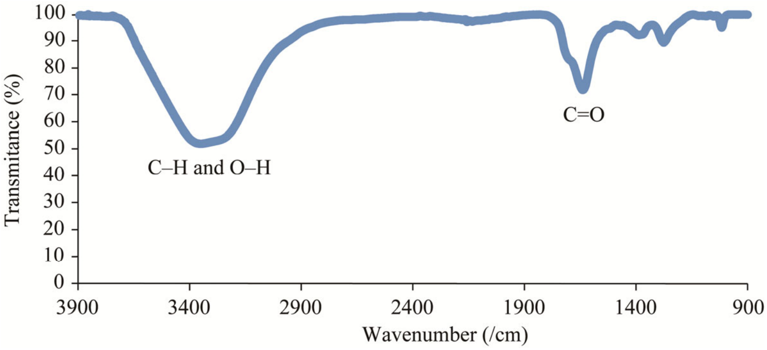

Fig. 1 Spectrum of the wood vinegar used in this study measured by Fourier-transform infrared spectroscopy (FTIR)

2.3 Kinetic experiment

To conduct a kinetic experiment on the release of the studied elements, we prepared a wood vinegar extract at a concentration of 1.00% wood vinegar solution from the wood vinegar sample. To accomplish this, we combined 1 mL of the mother wood vinegar solution with 100 mL of distilled water. Then, we suspended 2 g of dried soil samples in 20 mL of the extractant and agitated at 25°C using a reciprocating shaker for 30 min. Subsequently, we centrifuged the mixture at 3000 r/min, and added a fresh portion of the extractant to the remaining soil samples in the centrifuge tube and agitated the mixture for intervals of 1.0, 1.5, 2.0, 2.5, 3.0, 3.5, 4.0, 4.5, and 5.0 h. Following this, we determined the concentrations of Ca and Mg in all experimental time points through titration using EDTA and assessed the concentration of P using Olsen's method, which measures available P for plants (Olsen et al., 1954). To elucidate the kinetics of Ca, Mg, and P release from soil samples into wood vinegar solution, we employed four mathematical models (Table 2).

We calculated the coefficient of determination (R^2) using least squares regression and evaluated the standard error (SE) of the estimate using the appropriate formula:

$$\mathrm{SE} = \sqrt{\frac{\sum (X - X^*)^2}{n-2}}, \tag{1}$$

where X and X^* are the measured and calculated quantities of the element released at time t, respectively (mg); and n is the total number of data points.

We conducted the statistical analysis of the data using SPSS v.21.0 (IBM, Chicago, USA). To perform multiple comparisons of treatments, we employed analysis of variance (ANOVA) along with Tukey's Honestly Significant Difference (HSD) post hoc test, setting the significance level at $P<0.01$. Additionally, Microsoft Office Excel v.2019 (Microsoft, Redmond, USA) was utilized to create graphical representations.

Table 2 Mathematical formula of kinetics

Equation	Formula	Description	Reference
Elovich equation	$Y = a + b \ln t$	Y is the quantity of the element released at time t (mg/kg); a and b are constants, of which, a is the intercept of equation from the origin and b is the slope of equation; and t is the release duration (h).	Sparks (1989)
Parabolic diffusion law	$Y = a + b\sqrt{t}$		Havlin et al. (1985)
Power function equation	$\ln Y = \ln a + b \ln t$		Havlin et al. (1985)
Zero-order kinetics	$Y° - Y = a - bt$	$Y°$ is the maximum quantity of the element released at t time (mg/kg).	Martin and Spark (1983)

2.4 Mineralogy study

A representative sample within a similar physiographic unit from each cultivation was selected for X-ray diffraction (XRD) analysis. X-ray experiments were conducted both before and after treatment with the wood vinegar extractant. A mineralogical study was performed utilizing the soil fraction with particle size smaller than 2 mm. The soil samples were oven-dried at 100°C overnight, after which gypsum was removed through repeated washing with distilled water. Additionally, calcium carbonate ($CaCO_3$), organic matter, and iron oxides were eliminated using 1 N sodium acetate, 35.00% hydrogen peroxide (H_2O_2), and citrate-bicarbonate dithionate, respectively. The soil clay fractions were separated by centrifugation for 5 min at a speed of 750 r/min. The fraction smaller than 2 μm was subsequently treated with Mg-K saturation. Oriented slides were then prepared for further treatments, which included heating to 550°C and solvation with ethylene glycol. The samples were analyzed using an X-ray diffractometer XMD-300 (Unisantis Europe GmbH, Georgsmarienhütte, Germany). We performed the interpretation of the diffractograms according to the guidelines provided by Moore and Reynolds (1989), utilizing Xpowder v.2004 software (Daniel Martine, Granada, Spain).

3 Results

3.1 Soil physical-chemical properties

The properties of the soils under investigation are summarized in Table 3. The results indicated that all soil samples, except for the samples from sample site 5, exhibited a slightly alkaline pH. After a 5-h treatment with wood vinegar, the pH of all calcareous soils was measured and found to range from 6.00 to 6.40.

The highest CEC, measured at 20.21 cmol/kg, was observed in the soil associated with grape cultivation. Additionally, the highest CCE, recorded at 35.00%, was found in sample sites 1 and 3, which are also utilized for grape cultivation. All soils were classified as calcareous soils. The highest concentration of soluble Ca was recorded in grape cultivation, measuring 1002.03 mg/kg, while the lowest concentration was observed in rangeland, at 375.11 mg/kg. The maximum level of Mg was identified in grape cultivation, amounting to 180.05 mg/kg, whereas the minimum level was found in rangeland, at 45.10 mg/kg. Furthermore, the assessment of available P revealed the highest value in grape cultivation (17.00 mg/kg) and the lowest in rangeland (4.03 mg/kg). It can be asserted that the continuous cultivation of grapes, followed by wheat cultivation, has resulted in the release of additional nutrients from the soil, thereby increasing the available nutrient fraction. Variations in the levels of available P in soils may be attributed to differing application rates of P fertilizers, as well as the varying rates at which soluble forms of P are converted into less soluble forms.

Table 3 Physico-chemical properties of the soils under investigation

Land use	Sample site	Proportion (%)			pH	Content (mg/kg)			CEC (cmol/kg)	EC (dS/m)	Content (%)	
		Clay	Silt	Sand		Soluble Ca	Soluble Mg	Olsen P			OC	CCE
Vineyard	1	36.00	42.00	22.00	7.32	750.12	180.05	17.00	20.21	0.22	1.43	35.12
	2	24.00	50.00	26.00	7.34	1002.03	135.13	16.52	10.32	0.15	0.62	25.04
	3	24.00	28.00	48.00	7.42	820.05	165.32	15.04	19.52	0.15	0.91	35.32
Wheat field	4	21.00	18.00	61.00	7.91	675.22	120.05	7.62	11.64	1.42	2.43	21.23
	5	23.00	18.00	59.00	6.82	725.12	105.14	8.51	11.21	1.71	2.42	22.22
	6	31.00	9.00	60.00	7.63	530.22	75.11	6.52	10.00	1.81	2.00	18.61
Rangeland	7	22.00	15.00	63.00	7.13	450.06	90.13	4.03	7.81	1.52	2.82	16.11
	8	30.00	11.00	59.00	7.13	525.03	75.03	5.21	9.21	1.42	2.83	15.14
	9	24.00	7.00	69.00	7.24	375.11	45.10	6.00	10.81	1.42	2.64	26.12

Note: Ca, calcium; Mg, magnesium; CEC, cation exchange capacity; CCE, calcium carbonate equivalent. Olsen P means available P for plant.

3.2 Kinetics of element release from wood vinegar

3.2.1 Cumulative amount and duration of release

As illustrated in Table 4, the release of Ca ranged from a minimum of 28,800.00 mg/kg in rangeland to a maximum of 39,500.00 mg/kg in grape cultivation. Additionally, Mg release exhibited variability in all three cultivations, with values ranging from 2400.00 to 5880.00 mg/kg in grape cultivation. The cumulative P release in rangeland was observed to be the lowest, varying from 1.53 to 2.85 mg/kg. The comparative analysis of the average quantities of released elements across the three types of crop land indicated that there was no statistically significant difference in the amount of released Ca between grape and wheat crops (Fig. 2). However, a significant difference was observed between grape cultivation and rangeland use ($P<0.01$). Furthermore, the amount of accumulated Mg released exhibited a significant difference between grape and wheat cultivation, while no significant difference was noted in comparison to rangeland. The quantity of accumulated P released showed a statistically significant difference between grape cultivation and both wheat and rangeland land types ($P<0.01$). However, no significant difference was observed between wheat and rangeland land types.

Table 4 Release range of various elements using the wood vinegar in different types of crop land

Land use	Average cumulative release (mg/kg)		
	Ca	Mg	P
Vineyard	31,000.00–39,500.00^{a}	2400.00–5880.00^{a}	1.52–5.00^{a}
Wheat field	31,400.00–33,200.00^{ab}	2400.00–3540.00^{b}	1.65–2.75^{b}
Rangeland	28,800.00–31,400.00^{b}	3240.00–4500.00^{a}	1.53–2.85^{b}

Note: Different lowercase letters within the same colomn indicate significant differences among different types of crop land at $P<0.01$ level.

Figure 3 illustrates the release process of the desired elements across 10 consecutive time series. It was evident that grape cultivation was associated with the highest release rate among the three types of crop land studied. The trend in Ca release demonstrated an increasing pattern, suggesting that if the release period were extended, this trend would likely continue. The curve associated with Mg release exhibited a sustained trend characterized by a decreasing slope. Furthermore, the release rate of P was comparable to that of Ca, displaying a steeper slope.

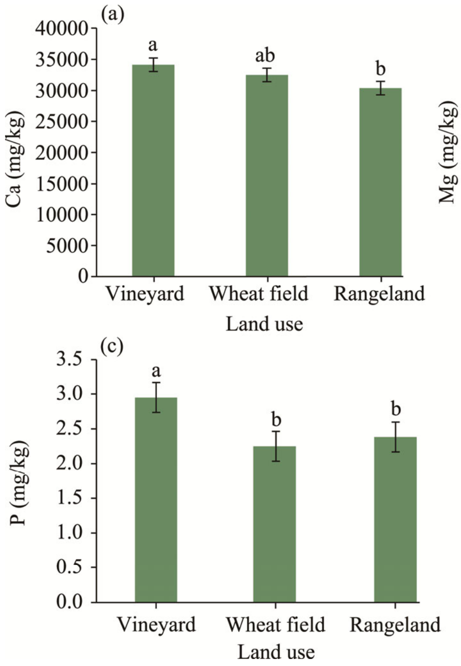

Fig. 2 Comparison of the average cumulative Ca (a), Mg (b), and P (c) released by wood vinegar in different cultivation lands. Ca, calcium; Mg, magnesium; P, phosphorus. Different lowercase letters indicate significant differences among different types of crop land at $P<0.01$ level.

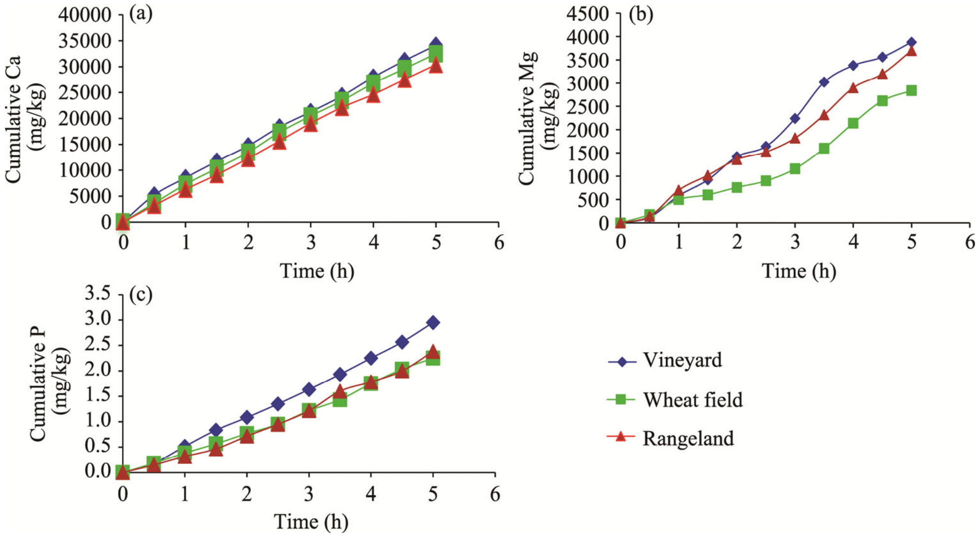

Fig. 3 Kinetics curve of Ca (a), Mg (b), and P (c) release from soil in different cultivation lands

A comprehensive comparison of the elements released by wood vinegar over a duration of 5.0 h, in relation to findings from other studies, indicated that the extraction capacity of wood vinegar surpassed that of citric acid and oxalic acid in calcareous soils as well as other land types. Selected comparisons are presented in Table 5. The data indicated that the quantities of Ca and Mg released by wood vinegar significantly exceeded those obtained from other extractants. Regarding of P, with the exception of 10 mM citric acid, the amounts of P released in other cases were reported to be lower than those from wood vinegar. It is important to consider that both the concentration of organic acids used and the duration of the extraction process play a crucial role in the extent of nutrient release.

Table 5 Comparison of elements released with different extractants

Extractant	Concentration	Duration	Cumulative release (mg/kg)			Reference
			Ca	Mg	P	
Acetic acid	10 mM	1.0 h	1078.00	2797.00	4.23	Etemadian et al. (2018)
Nitric acid	0.1 N	256.0 h	-	-	3.85	Sokhanvar Mahani et al. (2023)
Mixture of citric acid and oxalic acid	10 mM	5.0 h	2986.00	1728.00	-	Chahardoli (2022)
Ammonium acetate	1.0 N	60 d	-	239.37	-	Dash et al. (2023)
	1.0 N	1.0 h	1081.00	126.00	-	Senthurpandian et al. (2009)
Citric acid	10 mM	24.0 h	-	-	8.40	Lü et al. (2015)
Calcium chloride	10 mM	3.0 h	-	-	46.42	Jalali and Ahmadi Mohammad Zinli (2011)
Wood vinegar	1.00%	5.0 h	39,500.00	5880.00	5.00	This research

Note: -, no data.

3.2.2 Comparison of kinetic equations

Based on the R^2 and SE associated with the synthetic models employed, the zero-order kinetics demonstrated the most effective representation of the element release rates, followed by the power function equation, parabolic diffusion law, and, lastly, the Elovich equation.

As illustrated in Table 6, the highest R^2 (0.99) and the lowest SE (165.11 mg/kg) for Ca were associated with the zero-order kinetics in the context of grape cultivation. The highest R^2 (0.99) and the lowest SE (140.10 mg/kg) for Mg were associated with the zero-order kinetics in rangeland use. Similarly, grape cultivation was linked to a zero-order kinetics with R^2 of 0.99 and SE of 162.70 mg/kg. In contrast, wheat cultivation demonstrated a lower R^2 (0.95) and a higher SE (224.50 mg/kg) in its zero-order kinetics. For P element, the highest R^2 (0.99) and the lowest SE (0.04 mg/kg) were found in the zero-order kinetics for grape cultivation. Both wheat cultivation and rangeland exhibited an R^2 of 0.99; however, their SEs were higher, ranging from 0.06 to 0.08 mk/kg.

The constants of the kinetic equations used to describe the element release rates are presented in Table 7. The slope of equation (b) indicates the rate of interlayer element release, while the intercept from the origin (a) represents the initial release of element. The values of b, which serve as element release rates, are critical in equations that demonstrate the kinetics of element release. In this context, the maximum initial release amount of Ca, as determined by the zero-order kinetics, was recorded at 32,133.06 mg/kg in grape cultivation. The initial Ca release values across all three cultivation types, as described by the zero-order kinetics, ranged from 30,169.02 to 32,133.06 mg/kg. Furthermore, the highest initial release of Mg during the first half hour, similar to that of Ca in the zero-order kinetics, was also observed in grape cultivation, with a value of 4217.00 mg/kg. The highest concentration of P was also recorded in the zero-order kinetics during grape cultivation measured 3.11 mg/kg, with a variation range of 2.42–3.11 mg/kg across all three cultivation types.

Following the zero-order kinetics, the power function provided the most effective representation of element release rates across all three types of crop land. In the power function equation, the coefficient (b) of the extractant less than 1.00 suggests that the rate of element release decreases over time. The highest and lowest release rates associated with the power function equation in rangeland and grape cultivation, recorded at 0.98 and 0.83 mg/(kg•h) for Ca element. For the elements Mg and P, the highest release rates observed in grape cultivation and rangeland were recorded at 1.45 and 1.22 mg/(kg•h), respectively.

The analysis indicated that for the element Ca, the highest release rate, as described by the parabolic law, was associated with wheat cultivation, measuring 19,325.00 mg/(kg•h$^{1/2}$). The release rates for grape cultivation and rangeland use were 19,273.00 and 18,299.02 mg/(kg•h$^{1/2}$),

Table 6 Coefficient of determination (R^2) and standard error (SE) of estimation for kinetic equations used to assess the release rates of Ca, Mg, and P with wood vinegar extractant in different cultivation lands

Land use	Element	Zero-order kinetics		Power function equation		Parabolic law		Elovich equation	
		R^2	SE (mg/kg)	R^2	SE (mg/kg)	R^2	SE (mg/kg)	R^2	SE (mg/kg)
Vineyard	Ca	0.99	165.11	0.99	692.02	0.98	1534.01	0.91	3189.11
	Mg	0.99	162.72	0.97	337.31	0.97	259.43	0.89	463.24
	P	0.99	0.04	0.99	0.06	0.97	0.16	0.89	0.31
Wheat field	Ca	0.99	320.02	0.99	251.62	0.98	1319.04	0.92	2990.00
	Mg	0.95	224.50	0.97	242.00	0.88	345.22	0.77	477.31
	P	0.99	0.06	0.99	0.06	0.95	0.16	0.86	0.28
Rangeland	Ca	0.99	323.01	0.99	307.41	0.98	1328.90	0.91	2912.70
	Mg	0.99	140.10	0.96	178.03	0.95	263.05	0.87	431.32
	P	0.99	0.08	0.99	0.06	0.95	0.18	0.85	0.31

Table 7 Release rate of Ca, Mg, and P with wood vinegar extractant in different cultivation lands

Land use	Element	Zero-order kinetics		Power function equation		Parabolic law		Elovich equation	
		a (mg/kg)	b (mg/(kg•h))	a (mg/kg)	b (mg/(kg•h))	a (mg/kg)	b (mg/(kg•$h^{1/2}$))	a (mg/(kg•h))	b (mg/(kg•h))
Vineyard	Ca	32,133.06	6448.00	8699.01	0.83	10,887.01	19,273.00	9377.00	12,672.00
	Mg	4217.00	877.51	447.62	1.45	2093.01	2624.03	666.60	1724.02
	P	3.11	0.62	0.47	1.15	1.33	1.84	0.58	1.16
Wheat field	Ca	31,747.05	6444.01	7045.00	0.95	12,226.00	19,325.00	8057.01	12,749.02
	Mg	3152.02	579.33	391.53	1.15	1427.04	1735.02	424.61	1107.00
	P	2.42	0.53	0.38	1.10	1.32	1.41	0.42	0.89
Rangeland	Ca	30,169.02	6108.00	6311.03	0.98	12,042.03	18,299.02	7178.03	12,054.01
	Mg	3902.01	752.91	489.82	1.32	1684.00	2236.00	671.61	1463.00
	P	2.62	0.53	0.32	1.22	1.23	1.54	0.37	0.96

Note: a is the maximum release amount at initial 0.5 h; b is the release rate.

respectively (Table 7). In the context of Mg release rates, the highest observed rate, as described by the parabolic law, was associated with grape cultivation, measuring 2624.03 mg/(kg•h$^{1/2}$). The lowest rate, associated with the parabolic law in wheat cultivation, was recorded at 1735.02 mg/(kg•h$^{1/2}$). The highest rate of P release in grape cultivation, linked with a parabolic law equation, measured 1.84 mg/(kg•h$^{1/2}$). The lowest release rate of P, corresponding to a parabolic equation, was observed in wheat cultivation at 1.41 mg/(kg•h$^{1/2}$).

The results of the Elovich equations indicated that the highest rate of Ca release was 12,749.02 mg/(kg•h) in wheat cultivation, while the lowest rate was 12,054.01 mg/(kg•h) in pasture use. Additionally, the highest Mg release rate was observed in vineyard, measuring 1724.02 mg/(kg•h), whereas the lowest was recorded in wheat cultivation at 1107.00 mg/(kg•h). The maximum and minimum P release rates were observed in vineyard and wheat field, respectively, at 1.16 and 0.89 mg/(kg•h).

3.3 X-ray diffraction of clay sample

3.3.1 Before wood vinegar treatment

The semi-quantitative abundance of clay minerals in three soil samples collected from different cultivation sites is presented in Table 8. In the context of grape cultivation, it was clear that vermiculite clays, along with mixed vermiculite-illite and illite-smectite clays, were the predominant clay types. In contrast, smectite and illite clays were the most prevalent in wheat cultivation. Furthermore, the highest values related to range utilization were attributed to the presence of illite and vermiculite clays, followed by smectite. Consequently, due to the greater prevalence of vermiculite, illite, and smectite clays, as well as their mixed forms, significant changes and transformations were expected in these clays following the application of wood vinegar.

3.3.2 After wood vinegar treatment

The semi-quantitative analysis of clay mineral abundance in three soil samples, collected after treatment with wood vinegar, is presented in Table 9. The diffraction patterns of the clay samples, obtained following extraction with wood vinegar, indicated a significant alteration in mineral composition. The peaks corresponding to 14.50–15.00 Å (first order of vermiculite) and 7.00–7.50 Å (second order of vermiculite) have been completely eliminated (Fig. 4). The only peaks consistently observed across all three treatments were the 4.20 Å peak associated with quartz and the 3.30 Å peak related to feldspar. The changes in clay composition were comparable across all three applications, with no significant differences noted among the three types of crop land. A complete dissolution of all smectite clays, vermiculite, and palygorskite, as well as a lesser extent of illite, has been documented. The presence of a 5.00 Å peak, indicative of second-order illite, suggested its existence, albeit at a very low intensity. The residual 5.00 Å peak following the dissolution of the minerals was attributed to the presence of the mica mineral muscovite.

Table 8 Semi-quantitative analysis of clay mineralogy in soil samples from different types of crop land before the wood vinegar treatment

Sample site	Land use	Content rank of clay mineral
2	Vineyard	Vermiculite>mixed minerals>feldspar>smectite>illite>chlorite>kaolinite>quartz
5	Wheat field	Smectite>illite>chlorite>vermiculite>mixed minerals>palygorskite>kaolinite>quartz
8	Rangeland	Illite>vermiculite>smectite>mixed minerals>palygorskite>chlorite>kaolinite>quartz

Table 9 Semi-quantitative analysis of clay mineralogy in soil samples from different cultivation lands after wood vinegar treatment

Sample site	Land use	Content rank of clay mineral
2	Vineyard	Feldspar>quartz>mixed minerals>illite
5	Wheat field	Feldspar>quartz>mixed minerals
8	Rangeland	Feldspar>quartz

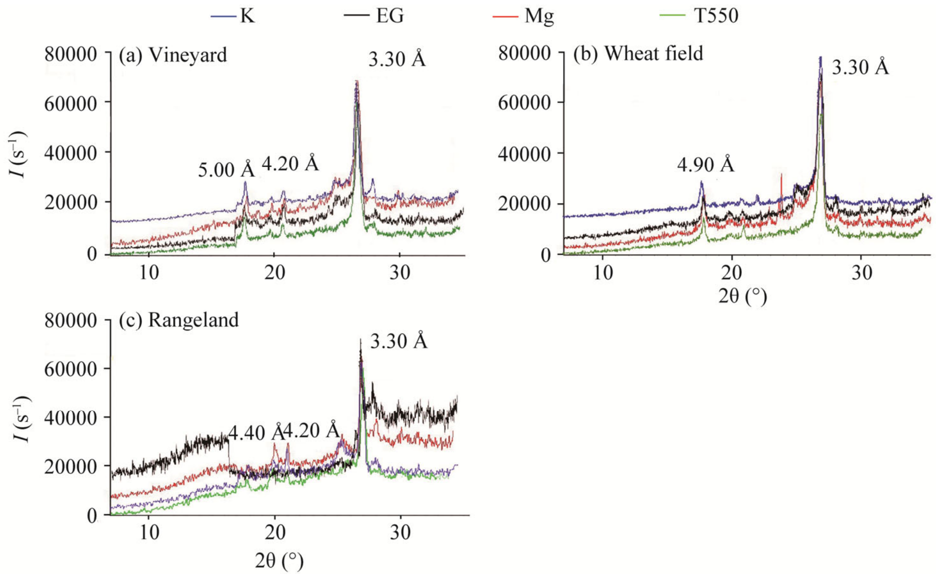

Fig. 4 X-ray diffraction graph for the soils sampled from vineyard (a), wheat field (b), and rangeland (c) after wood vinegar treatment. EG, ethylene glycol; T550, temperature at 550°C.

4 Discussion

Based on the findings, it was determined that wood vinegar, an organic acid, releases significant amounts of Ca, Mg, and even P, particularly in grape cultivation. The primary factor contributing to this release is the reduction of soil pH to 6.00–6.40 after a 5-h treatment with wood vinegar. Hydronium ions (H_3O^+) released from acetic acid in wood vinegar can dissolve soil carbonates, thereby facilitating the release of Ca and Mg (Golubev et al., 2006). Etemadian et al. (2018) demonstrated that organic acids have a more substantial influence on the release of elements, including Ca, Mg, and P, compared with mineral acids. They reported that the primary factor contributing to this phenomenon was a decrease in soil pH, which ranged from 0.03 to 1.66. The increased clay content (rising from 24.00% to 36.00%) and silt content (ranging from 28.00% to 50.00%) in grape cultivation contributed to the enhanced release of essential elements from the soil. These textural changes are particularly important for enhancing P release in grape cultivation (Gou et al., 2020). Additionally, increasing the silt content has proven effective in promoting Mg release (Simard et al., 1992). Mineralogical analyses of the region's soils revealed the presence release (Simard et al., 1992). Mineralogical analyses of the region's soils revealed the presence of vermiculite, smectite, and palygorskite clays, which serve as sources of Mg and Ca (Hashemi, 2020). Etemadian et al. (2018) reported that the highest concentration of P released, initially, facilitated by phosphoric acid and subsequently by acetic acid, was measured at 4.23 mg/kg within 1.0 h. Furthermore, the application of wood vinegar has been shown to reduce soil pH (Najafi-Ghiri et al., 2024), which subsequently enhances the availability of P (Osori and Mix, 2016). Consequently, the highest concentrations of Ca, Mg, and P were recorded in grape cultivation. Following the addition of wood vinegar to calcareous soil samples, soil acidity was observed to range from an average pH of 6.00 to 6.40. Other studies have reported similar results when utilizing wood vinegar (Yatagai et al., 2002; Golubev et al., 2006; Najafi-Ghiri et al., 2024). Additionally, a study examining various crops—including potatoes, garlic, vegetables, and wheat—as well as rangeland use, demonstrated that the rate of P release varied among different crops; however, these differences were not statistically significant. The highest P release was observed in potato cultivation, while the lowest was recorded in rangeland management (Jalali and Ahmadi Mohammad Zinli, 2011).

After 5.0 h, the amount of released Ca has not yet reached an equilibrium state, indicating that a longer duration is necessary for the release of additional elements (Etemadian et al., 2018). Senthurpandian et al. (2009) demonstrated that the amount of releasable Ca by ammonium acetate ($NH_4CH_3CO_2$) decreased by approximately 40.00%–50.00% during the second extraction compared with the first extraction across nearly all soil samples. They reported that the constant release of Ca was observed only after the eighth extraction. In wood vinegar, after ten extraction cycles, Ca has not yet reached a steady release. This indicates that the extraction process will continue. The curve associated with the release of Mg exhibited a continuous trend characterized by a reduced slope. The rate of release was significantly lower in comparison with that of Ca; however, it did not reach an equilibrium. It can be posited that acetic acid may facilitate the release of Mg ions from soil clay by weakening the bonds between Mg and the mineral matrix (Srinivasarao et al., 2006; Chahardoli, 2022). Dash et al. (2023) investigated Mg release and found that, during the initial stages, the release exhibits a steeply inclined curve over brief time intervals, indicating a substantial increase in Mg mass within a short duration. In the subsequent stage, the slope of the Mg release curves decreases, resulting in a linear profile that is nearly parallel to the *y*-axis. A study conducted by Mohammad Jafari et al. (2015) demonstrated that the rate of Mg release from sepiolite mineral is positively correlated with the concentration of organic acids. Furthermore, it was observed that the rate of Mg release increases as the pH of the environment decreases (Najafi-Ghiri et al., 2024) and as the particle size of the sepiolite mineral diminishes (Simard et al., 1992; Gou et al., 2020). It can be concluded that the reduction in pH, resulting from the addition of wood vinegar, has facilitates the release of Mg from clay minerals that contain this element, such as vermiculite and palygorskite. The release rate is notably high during the initial stages but subsequently decreases (Srinivasarao et al., 2006; Senthurpandian et al., 2009; Mohammad Jafari et al., 2015; Chahardoli, 2022). The dissolution of Mg carbonates by hydrogen ions (Senthurpandian et al., 2009) and the complexation of Mg in minerals by acetic acid in wood vinegar are possible (Golubev et al., 2006). The dissolution of carbonates occurs due to the enhanced ability of an acid to release hydrogen ions. Both of these factors contribute to the increased concentrations of Ca and Mg observed in the release analysis. The maximum cumulative P release was observed in grape cultivation. Sokhanvar Mahani et al. (2023) demonstrated that the highest P release in soil treated with acidified vermicompost, after a duration of 256.0 h, was measured at 3.85 mg/kg. This phenomenon was attributed to a decrease in pH levels. Additionally, Yang et al. (2019) reported that the release of P facilitated by organic acids, specifically oxalic and citric acids, occurred rapidly at first, followed by a slower release phase that extended up to 2160.0 h. Singla Just et al. (2024) conducted a 160 d soil incubation study to investigate the release of P from various types of ashes. Their findings revealed that all tested ashes released over 50.00% of the total applied P within the initial 5 to 10 d. The application of wood vinegar effectively lowered the pH of the lime soils under investigation resulting in enhanced solubility and subsequent release of P, which is attributed to the enhanced solubility of iron and aluminum phosphates, as well as calcium phosphate (Wang et al., 2017). Consequently, it can be concluded that the buffering capacity of calcareous soils inhibited any further reduction in soil pH. Jalali and Ahmadi Mohammad Zinli (2011) reported that the rapid release of P in the early phases can be attributed to the swift dissolution of amorphous phosphates with low bond energy.

The results of this study suggested that the most suitable model for Ca and Mg was associated with zero-order kinetics. This result was corroborated by the results of other researchers (Senthurpandian et al., 2009; Chahardoli, 2022). Following the zero-order kinetics, the power function equation was determined to be the most suitable for representing the release of elements (Chahardoli, 2022; Dash et al., 2023). In the context of P, the model that provided the best fit was represented by a zero-order kinetics following power function equation (Yang et al., 2019; Sokhanvar Mahani et al., 2023). The results of the zero-order kinetics indicated that acetic acid in wood vinegar was effective in degrading minerals during short-term experiments (Ström et al., 2005). However, it was found to be more effective in soils with higher contents of carbonate

(Senthurpandian et al., 2009; Najafi Ghiri et al., 2024) and clay minerals (Mohammad Jafari et al., 2015), such as vermiculite, palygorskite, and smectite, which are commonly observed in grape cultivation. The dissolution and destruction of clay minerals with wood vinegar may be attributed to the complexation of aluminum and Mg with acetate (Ström et al., 2005). The dissolution of clay minerals, as well as their degradation by organic acids, has been documented in numerous kinetic studies, highlighting the availability of certain nutrients in accessible forms (Srinivasarao et al., 2006; Jenagh et al., 2015; Li et al., 2021; Dash et al., 2023; Hashemi and Najafi-Ghiri, 2024).

The results indicated that the initial release amount (a) of the zero-order kinetics was the highest (Heshmati et al., 2023), while the diffusion parabolic equation represented the highest release rate (b) (Chahardoli, 2022; Hashemi and Najafi-Ghiri, 2024). The high release rate in the parabolic diffusion law indicated that the primary factor influencing element release is their diffusion from the mineral matrix (Havlin et al., 1985). The elevated release rate can be attributed to the increased dissolution of lime in both grape and wheat crops due to the resulting acidity (Sokhanvar Mahani et al., 2023). Initially, mineral dissolution and the rapid diffusion of the element occurred; however, over time, this diffusion rate decreased (Srinivasarao et al., 2006). Grape cultivation exhibited the highest rate of Mg release. The higher b constant in grape culture indicated the release of Mg from the non-exchangeable portion of minerals (Havlin et al., 1985). The high content of vermiculite, smectite and palygorskite clays in grape cultivation may primarily account for the increased Mg release rate in these soils. Grape soils with a higher content of smectitic and palygorskite clays, as well as edge and wedge sites, exhibited greater release rate constants (Srinivasarao et al., 2006). Furthermore, Chahardoli (2022) reported a similar trend, identifying the maximum Mg release rate in the parabolic diffusion law with $CaCl_2$ as the extractant. Also, the kinetic analysis of P indicated that the parabolic equation exhibited the highest release rate in grape cultivation. The release rate constants of the parabolic diffusion law are influenced by the diffusion gradient between the interlayer and exchange surfaces within the mineral structure. The diffusion law is based on the assumption that the rate-limiting step is the diffusion of P ions from the interior of the particle to its surface (Srinivasarao et al., 2006; Singla Just et al., 2024). Furthermore, the application of large quantities of organic P fertilizers in grape cultivation, combined with their stabilization in calcareous soils, has rendered P becoming unavailable. Additionally, acidifying the soil with wood vinegar enhances the solubility and availability of P in calcareous soils (Sokhanvar Mahani et al., 2023). The observed range of P fluctuations in the zero-order kinetics was relatively extensive, and these findings aligned with the results reported by other researchers (Etemadian et al., 2018; Sokhanvar Mahani et al., 2023). Therefore, it can be asserted that the influence of wood vinegar on the release of the aforementioned elements was more pronounced in grape culture soils.

The X-ray results indicated that the application of wood vinegar to soil has led to the elimination of peaks associated with various clay minerals. Despite the soil's buffering capacity, which decreased the pH of soil samples to 6.00–6.40 following treatment, the presence of acetic acid in wood vinegar, along with its low initial pH, contributes to the degradation of clay minerals (Li et al., 2021). The absence of the 14.50–15.00 Å and 7.00–7.50 Å peaks suggested that the vermiculite clay has been completely degraded. The detection of the 5.00 Å peak in the low-intensity Mg saturation treatment indicated the presence of trioctahedral mica minerals (Fanning et al., 1989). The only mica mineral that remained stable in the presence of acidic solutions was dioctahedral mica, specifically muscovite (Pal et al., 2001; Melo et al., 2002). Research has demonstrated that sequential extraction of K and Mg using citric acid over 15 cycles, leads to the dissolution of trioctahedral minerals found in the silt and clay fractions of soil. This process subsequently transforms dioctahedral mica minerals into vermiculite (Simard et al., 1992). The application of acidic conditions leads to the release of Mg and the transformation of montmorillonite into kaolinite. Additionally, the concentrations of released iron and Mg suggest a random distribution of these ions within the octahedral sheet of montmorillonite (Li et al., 2021). The 4.20 Å peaks associated with quartz and the 3.30 Å peaks corresponding to feldspar remained stable across all three treatments. Furthermore, the stability of the feldspar peak indicated that the release of K from this mineral has not yet been completion. It has been documented that the application of wood vinegar enhances the levels of dissolved K and Ca in the soil, a phenomenon attributed to the dissolution of acetic acid and the release of K from mineral sources (Srinivasarao et al., 2006). Yin et al. (2023) demonstrated in their research that the type and quantity of clay minerals can be influenced by acidity. They found that montmorillonite is predominantly present in environments with weak acidity, while gibbsite is more prevalent in strongly acidic conditions. Furthermore, they noted that when acidity levels drop below 4.50, clay minerals are completely hydrolyzed and converted into gibbsite. Consequently, it can be concluded that the acidity induced by wood vinegar is a significant factor influencing the transformation and degradation of clay minerals in the soil.

5 Conclusions

This study examined the impact of wood vinegar as an organic extract on the release kinetics of Ca, Mg, and P in three distinct crops grown in calcareous soils. The findings indicated that the addition of wood vinegar over a period of 5.0 h effectively reduced the acidity of the calcareous soils, thereby enhancing the dissolution of minerals and increasing the availability of all three elements. However, the release rate has not yet reached equilibrium, necessitating a longer duration to further enhance the release and maintain stability. The most substantial effect of wood vinegar on the release of all three elements was observed in grape cultivation. Additionally, the zero-order kinetics demonstrated the best fit for all three elements. The results of the zero-order kinetics indicated that wood vinegar was effective in breaking down minerals in short-term experiments. It was particularly effective in soils with higher concentrations of carbonate and clay minerals, such as vermiculite, palygorskite, and smectite, as observed in grape cultivation. Under the acidic conditions induced by the addition of wood vinegar, vermiculite, smectite, and palygorskite minerals were completely destroyed, while illite was partially dissolved. Furthermore, considering the field's focus on the release of elements, it is crucial to assess the changes in essential elements influenced by cultivation over an extended period, both in the soil and in the plants. Due to its pesticide properties, wood vinegar is recommended for improving the availability of essential nutrients in calcareous soils. The findings indicated that the highest efficacy from the application of this organic compound is observed in grape cultivation, suggesting a potential reduction in reliance on chemical fertilizers in agricultural practices and orchards, which could result in cost savings.

Conflict of interest

The author declares that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Author contributions

Conceptualization, data curation, methodology, formal analysis, writing - original draft preparation, writing - review and editing, and funding acquisition: Soheila Sadat HASHEMI. The author approved the manuscript.

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