Effects of Torrefied Sawdust–Vermiculite Mixtures as Ethylene Scavengers on Shelf-Life Extension of Fruit Quality

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Torrefied sawdust has gained attention as a carbon-rich material with enhanced porosity and adsorption properties, making it a promising candidate for ethylene scavenging to extend fruit shelf life. In this study, torrefied oak sawdust was mixed with vermiculite to prepare ethylene scavenger mixtures, and their adsorption performance and effects on fruit quality were systematically evaluated. Laboratory-scale measurements showed that increasing the proportion of torrefied sawdust significantly enhanced ethylene adsorption. Response surface methodology (Box–Behnken design) identified optimal preparation conditions (66% torrefied sawdust, 2 mm particle size, and 342 h exposure) yielding an adsorption efficiency of approximately 84%. Compared with pure vermiculite, the optimized mixture provided substantially higher ethylene removal capacity and contributed to more than a two-fold extension of apple shelf life during storage. These findings highlight the potential of torrefied sawdust–based mixtures as effective, low-cost ethylene scavengers for postharvest fruit preservation and provide valuable design parameters for future material optimization.

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INTRODUCTION

Torrefied sawdust is a carbon-rich material produced by heating biomass typically within the range of 250 to 350 °C in an oxygen-limited environment, resulting in enhanced porosity and gas adsorption characteristics. It has several advantages, including a large specific surface area, a pore structure that favors gas adsorption, and low density (Kadzere *et al*. 2002; Yağcı *et al*. 2024). Some early studies have shown that heated sawdust can delay ripening of fruits and improve their postharvest quality (Matan and Songsamoe 2022). It is more likely that the torrefied sawdust reduces ethylene levels by adsorbing the gas from the surrounding air, thereby limiting its availability to trigger fruit ripening (Spokas *et al*. 2010). But the mechanisms affecting this process remain unclear. Recently, the effect of binding heated sawdust *in vitro* was reported (Di Lonardo *et al*. 2013), but the effect on ethylene biosynthesis was not clear.

During torrefaction, typically conducted between about 250 and 350 °C under oxygen-limited conditions, lignocellulosic biomass undergoes selective thermal degradation of hemicellulose, partial deoxygenation of cellulose, and structural modification of lignin (Martín *et al*. 2022). These chemical changes lead to a decrease in oxygen- and hydrogen-containing functional groups, an increase in the relative carbon content, and the development of more aromatic and thermally stable structures, which together modify the porosity and surface chemistry of the treated wood (Zhang *et al*. 2025).

Such torrefied materials and biochars have been widely reported as promising adsorbents because of their enhanced surface area, pore volume, and tunable surface functional groups (Tan *et al*. 2021). They have been applied for the adsorption of gases such as CO_2 and various organic or inorganic pollutants (Ambaye *et al*. 2021). Although direct studies on ethylene adsorption by torrefied wood are limited, previous work on biochar–plant systems has indicated that carbonized woody materials can affect ethylene dynamics in the surrounding environment, suggesting their potential as ethylene-modulating or scavenging materials (Das *et al*. 2022). In this context, the present study aimed to systematically evaluate the ethylene adsorption efficiency of torrefied sawdust–vermiculite mixtures and to optimize their preparation conditions for extending fruit shelf life.

While apples are a popular fruit with a high demand in most countries, they ripen relatively quickly after harvest, causing them to change color and soften. This is an important problem that can affect both the consumption and sale of apples. Therefore, there is a need for technologies to inhibit or adsorb ethylene gas, which is the main cause of rapid oxidation of apples, and to increase the time to ripening (Zhou *et al*. 2000). Plant growth regulators and related chemical treatments that affect the growth and ripening of fruits have been used to inhibit spoilage during the storage period (Sebastian *et al*. 2019). However, there are limitations in terms of increasing the storage period of apples and not compromising their quality (Zhong *et al*. 2018). Ethylene is a plant hormone involved in the ripening of most fruits or vegetables (Sharma *et al*. 2012). It plays an important role in shortening the storage period of apples (Hertog *et al*. 2016). Therefore, scavengers for ethylene adsorption utilizing different substrates have been investigated, which have helped to improve the quality of various fruits (Fan *et al*. 2000). Apples are climacteric fruits whose ripening is tightly regulated by ethylene, a plant hormone that triggers a surge in respiration, cell wall softening, pigment degradation, and aroma compound biosynthesis. As ethylene accumulates in the storage environment, it reinforces a positive feedback loop that accelerates further ethylene production in the fruit. By adsorbing and lowering the surrounding ethylene concentration, an ethylene scavenger disrupts this feedback signal, thereby slowing physiological ripening responses and extending shelf life.

Although previous studies have reported that heated or carbonized woody materials can influence ethylene-related processes in plant systems, there is a lack of research that directly quantifies ethylene adsorption by torrefied sawdust. Moreover, no published work has systematically optimized the preparation conditions (mixing ratio, particle size, and exposure time) of torrefied sawdust–based mixtures to maximize ethylene scavenging efficiency. The effect of such mixtures on the actual shelf-life extension of fruits has also not been previously evaluated. Therefore, the present study offers a novel contribution by providing, to our knowledge, the first quantitative assessment of ethylene adsorption efficiency of torrefied sawdust mixtures and their impact on delaying ripening during fruit storage.

Vermiculite, a lightweight silicate mineral with high water-holding capacity and moderate porosity, is widely used in horticultural and storage applications as a physical filler material. In ethylene control systems, vermiculite often serves as a baseline substrate because it exhibits limited but measurable adsorption capacity. In this study, vermiculite was used as a comparative reference to evaluate the enhanced adsorption performance of torrefied sawdust mixtures.

The authors stored apple fruits with different input weight of heated sawdust. Specifically, torrefied sawdust was used, and the adsorption of ethylene for apple fruits treated during storage or delivery was evaluated. The aim was to reveal a possible way that torrefied sawdust affects ethylene adsorption in apples.

EXPERIMENTAL

Preparation of Torrefied Sawdust

Oak sawdust was selected as a raw material to produce torrefied sawdust through a physicochemical method. The sawdust, obtained from the forest research site of Gyeongsang National University, was air-dried, crushed, and cut to an average particle size of 5 cm × 5 cm. The torrefied sawdust was produced in a fixed bed reactor at 330 °C under an oxygen-limited environment with a residence time of 15 minutes. The resulting product was then sieved to obtain particles in the 1 to 4 mm size range.


Non-treated sawdust

1 mm torrefied sawdust

2 mm torrefied sawdust

4 mm torrefied sawdust

Fig. 1. Torrefied sawdust as grinding size

Characterization of Torrefied Sawdust

The bulk density of the torrefied sawdust samples was analyzed by filling a 25-mL cylinder to a particular volume with a torrefied sawdust sample and determining its weight at this volume. Samples were immediately oven-dried at 105 °C for 24 h. The cylinders were respectively tapped for 10 min to densify the torrefied sawdust before measuring weight (no additional pressure or mechanical compaction was applied). The porosity of the torrefied sawdust was calculated following Eq. 1,

$$\text{Porosity (\%)} = (1 - B\,/\,T\,) \times 100 \tag{1}$$

where B is the bulk density of the sample (g/cm^3), and T is the true density of the sample (g/cm^3).

Fruit Materials

The apples were taken from trees growing well in South Korea in the summer of 2023, at their peak but mature stage. They were selected to ensure that they were uniform in various characteristics, including size, color, and maturity. The apples used in this study were the cultivar ‘Fuji’, which is one of the most widely grown apple varieties in South Korea and exhibits consistent ethylene production characteristics during storage.

Measure Method of Ethylene Adsorption

The ethylene concentration was measured using a custom-built detection system developed in the authors’ laboratory. To evaluate the ethylene adsorption effect of torrefied sawdust, vermiculite was used as the default material. In addition to the three mixture ratios (1:3, 1:1, and 3:1), a control condition using 0% torrefied sawdust (pure vermiculite) and a 100% torrefied sawdust condition were also included to provide baseline and upper-limit reference points for evaluating adsorption efficiency. The mixed substrates (torrefied sawdust and vermiculite) were packaged in breathable Tyvek material for manufacture into scavengers, respectively. It should be noted that the Tyvek material was used only as a permeable pouch to hold the scavenger mixture inside the chamber, while the ethylene adsorption test itself was conducted within a tightly sealed plastic bin, ensuring that ethylene could not escape during measurement. The ethylene adsorption test was conducted in a sealed plastic chamber equipped with two rubber hose ports. One port was connected to a cylinder supplying ethylene gas at a controlled pressure of 1 bar, an initial concentration of 1000 ppm, and a flow rate of 0.1 L/min. The torrefied sawdust–vermiculite mixtures, enclosed in breathable Tyvek pouches, were placed at the center of the chamber. The second port was connected to a calibrated gas detector to monitor the ethylene concentration exiting the chamber. All joints were tightly sealed with laboratory-grade tape to prevent gas leakage and ensure that ethylene passed exclusively through the scavenger materials during measurement. The ethylene adsorption experiment was performed for 1 h. The percentage of ethylene adsorption was calculated using Eq. 2,

$$\text{Ethylene oxide adsorption (\%)} = (T_i - T_e) / T_i \times 100 \tag{2}$$

where T_i is the concentration of ethylene gas initially injected, and T_e is the concentration of ethylene gas measured at the rubber hose through which the ethylene gas escapes. The ethylene concentration was expressed in units of mg/L. The ethylene adsorption test was carried out a total of three times.

Response Surface Methodology (RSM) Using Box–Behnken Design

The BBD utilized the ethylene adsorption efficiency for optimization. This design is a RSM suitable for three factors, and is presented as three levels (coded -1, 0, +1) (Table 1).

Table 1. Codes and Actual Levels of the Independent Variables for the Design of the Experiment

Independent Variables	Symbols	Code Levels		
		-1	0	1
Torrefied sawdust mixed ratio	%	0 (100% vermiculite)	100	50
Torrefied sawdust size	mm	1.0	2.5	4.0
Ethylene absorption period	h	48	360	672

The RSM analysis method is ideal for predicting maximum target numbers with relatively little experimental data. It requires only three factors and three ranges for each of those factors, while still providing a reliable statistical design. Therefore, this study was also designed with three factors and three ranges for each factor and consisted of three replicates of points at the midpoint of each corner of the multidimensional cube, for a total of 17 experiments. The 17 tests designed with BBD are shown in Table 2 along with the experimental data. The midpoint (Code Levels 0,0,0) was intentionally repeated three times in the experimental matrix. This repetition is a standard feature of Box–Behnken designs and is used to obtain an estimate of pure error, to test for curvature in the response surface, and to ensure the statistical robustness of the model fit. The results of the BBD experiments were fitted to a second-order polynomial equation using a multiple regression approach. The reliability of the predicted model was indicated by the coefficient of determination, R^2. Statistical significance was confirmed by F-value and *p*-value. The software used for the analysis was Statistica (version 5.0) from StatSoft, Inc. (Tulsa, OK, USA).

Statistical Analyses

Data were expressed as mean ± standard deviation (n = 3). Statistical analysis was performed at a significance level of 5% using Statistical Analysis System software (SAS Institute, Inc., Cary, NC, USA). Differences between group means were evaluated using Duncan's multiple range test using SAS.

Table 2. The Box-Behnken Response Surface Design and Corresponding Response Values

Run	Torrefied Sawdust Mixed Ratio (%)	Torrefied Sawdust Size (mm)	Ethylene Absorption Period (h)	Ethylene Adsorption Efficiency (%)
1	50	2.5	360	83.24
2	50	4.0	672	70.68
3	0	2.5	672	55.49
4	100	2.5	48	64.71
5	50	2.5	360	84.29
6	50	2.5	360	84.81
7	50	2.5	360	80.10
8	100	4.0	360	68.58
9	0	1.0	360	52.35
10	50	1.0	48	62.82
11	100	1.0	360	75.39
12	0	4.0	360	47.12
13	0	2.5	48	43.97
14	50	2.5	360	82.72
15	50	1.0	672	73.29
16	50	4.0	48	60.73
17	100	2.5	672	78.01

RESULTS AND DISCUSSION

Properties of Mixtures Based-torrefied Sawdust

This section aims to characterize the physical properties (bulk density, true density, and porosity) of the torrefied sawdust–vermiculite mixtures in order to understand how their structural features influence ethylene adsorption. Since porosity and density directly determine gas contact area and diffusion pathways, these measurements provide essential mechanistic context for interpreting the ethylene scavenging performance evaluated in subsequent experiments. The porosity, volume, and bulk density values of the torrefied sawdust samples are shown in Table 3. The bulk density is closely related to the porosity. Low bulk density can promote high porosity, and from the results, it can be confirmed that the torrefied sawdust helps to increase the porosity. The low bulk density suggests a relatively open packing structure of the torrefied sawdust, which may facilitate gas accessibility. However, it does not directly indicate the presence of adsorption-active micro- or mesopores. Therefore, it was speculated that the torrefied sawdust is effective in adsorbing ethylene gas from the many pores, *i.e.*, an increase in the mixing ratio weight of torrefied sawdust provides higher mechanical strength than vermiculite. As shown in Table 3, the bulk density of torrefied sawdust is suitable for use in ethylene scavenger mixtures. Almost all 4 mm torrefied sawdust samples have porosities greater than 80%. With respect to the particle size, the “mm” represents the mesh size through which the material was passed, with 1 mm, 2 mm, and 4 mm corresponding to over 50%, 70%, and 80% of the sample, respectively.

Figure 2 shows the porosity curves of the samples, with 90% of the 1 mm torrefied sawdust having a porosity of less than 60%, suggesting that the smaller particles of the resulting torrefied sawdust can penetrate the vermiculite space and reduce porosity. Previous studies have shown that torrefaction alters the physical properties of lignocellulosic biomass, including increases in BET surface area and pore development due to devolatilization and partial carbonization (Chen *et al*. 2017). These structural changes enhance the adsorption capacity of torrefied materials for gases such as CO_2 and various volatile compounds. Given this relationship, it is reasonable to propose that ethylene adsorption by torrefied sawdust–based mixtures may also be strongly influenced by BET surface area, although further work would be required to quantify this relationship.

Table 3. Properties of Mixtures Based-torrefied Sawdust

No.	Particle Size (mm)	Torrefied Sawdust (g)	Vermiculite (g)	Bulk Density (Mg/m^3)	True Specific Gravity (Mg/m^3)	Porosity (%)
1	-	0 g	4 g	1.15	4.34	73.46
2	-	0 g	7 g	0.12	0.49	75.57
3	-	0 g	14 g	0.09	0.38	75.76
4	1 mm	1 g	3 g	0.13	0.40	68.99
5		2 g	5 g	0.17	0.43	61.78
6		4 g	10 g	0.16	0.39	59.88
7		2 g	2 g	0.14	0.31	54.11
8		3.5 g	3.5 g	0.16	0.33	51.41
9		7 g	7 g	0.11	0.25	54.63
10		3 g	1 g	0.16	0.29	46.72
11		5 g	2 g	0.13	0.26	49.17
12		10 g	4 g	1.15	1.95	41.05
13		4 g	0 g	0.27	0.51	46.03
14		7 g	0 g	0.49	0.92	46.97
15		14 g	0 g	0.47	0.91	48.63
16	2 mm	1 g	3 g	0.30	0.91	67.37
17		2 g	5 g	0.28	0.91	69.39
18		4 g	10 g	0.13	0.41	67.63
19		2 g	2 g	0.16	0.52	69.43
20		3.5 g	3.5 g	0.16	0.49	67.55
21		7 g	7 g	0.15	0.50	69.68
22		3 g	1 g	0.15	0.52	70.66
23		5 g	2 g	0.15	0.50	70.39
24		10 g	4 g	0.13	0.45	71.91
25		4 g	0 g	0.14	0.61	77.94
26		7 g	0 g	0.09	0.44	79.38
27		14 g	0 g	0.09	0.41	77.60
28	4 mm	1 g	3 g	0.13	0.58	78.22
29		2 g	5 g	0.10	0.54	80.57
30		4 g	10 g	0.14	0.70	80.61
31		2 g	2 g	0.10	0.65	84.02

32		3.5 g	3.5 g	0.12	0.97	87.18
33		7 g	7 g	0.13	0.90	85.75
34		3 g	1 g	0.13	0.85	85.17
35		5 g	2 g	0.08	0.60	85.77
36		10 g	4 g	0.18	1.45	87.30
37		4 g	0 g	0.17	2.11	91.90
38		7 g	0 g	0.17	1.90	91.04
39		14 g	0 g	0.11	1.15	90.26

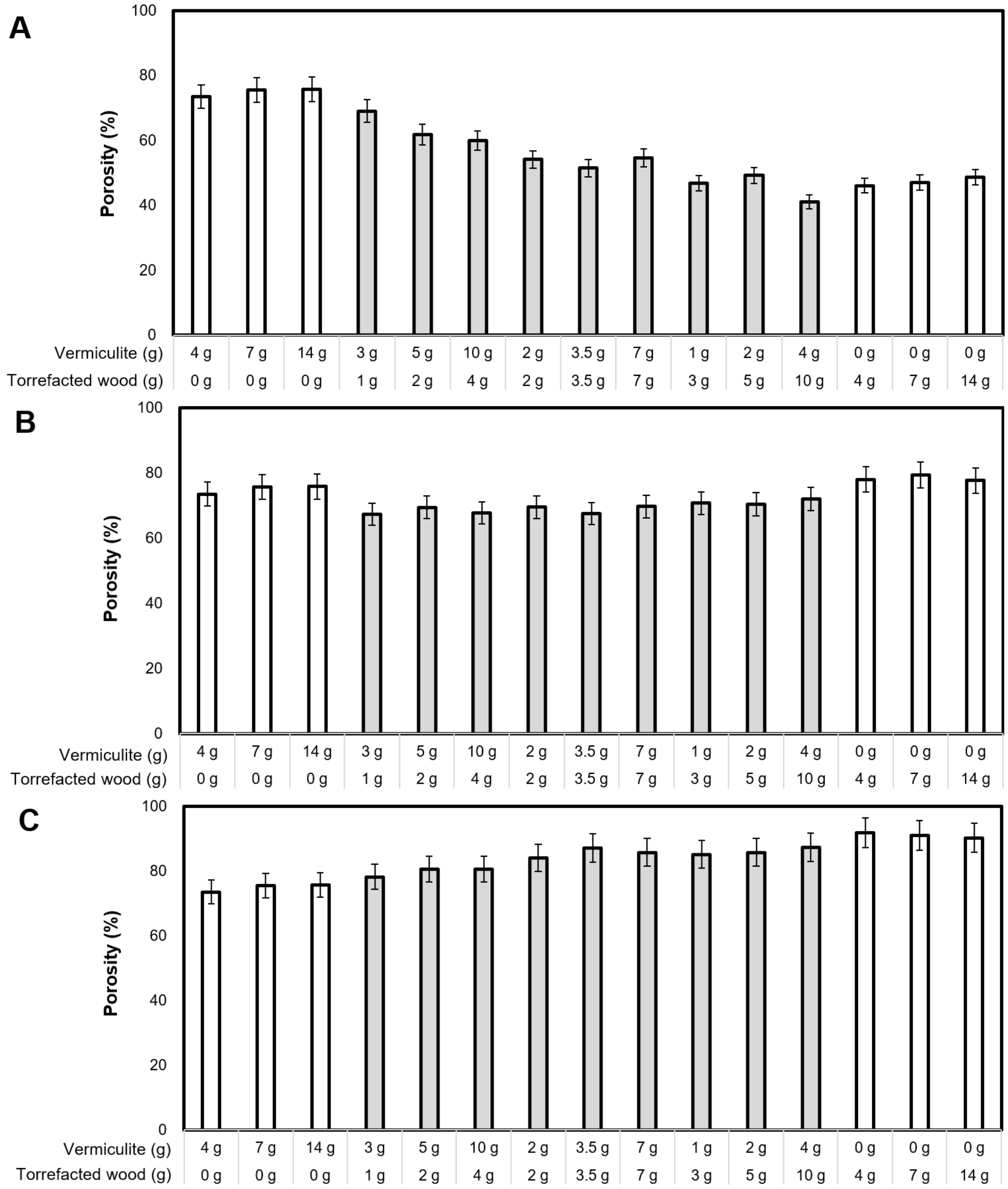
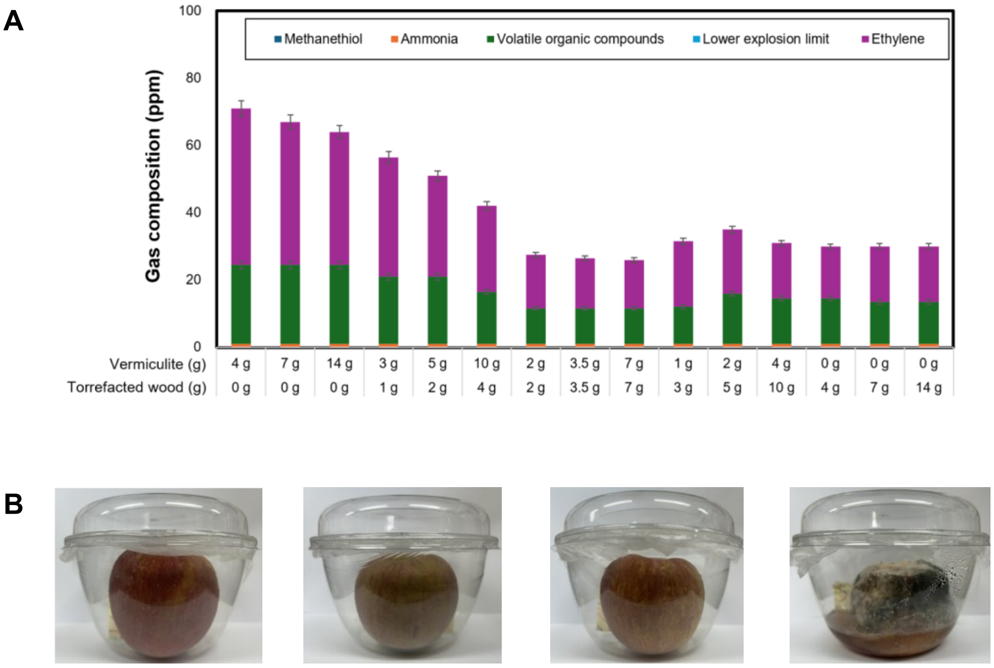


Fig. 2. Bulk density and gasification of mixtures based-torrefied sawdust A: 1 mm torrefied wood; B: 2 mm torrefied sawdust; C: 4 mm torrefied sawdust

Laboratory Scale Ethylene Adsorption Results

Figure 3 shows the ethylene adsorption efficiency of torrefied sawdust and vermiculite mixtures with varying additions of activated carbon. A laboratory-scale experiment was conducted to measure the adsorption efficiency of ethylene that passed through the system over a 28-day period. The experiment was conducted 5 times under constant conditions. The ethylene that passed through the preparation tools over the 28 days was found to be around 14 to 47 ppm. The torrefied sawdust mixtures showed good efficiency for ethylene adsorption, especially for the mixture containing 50% of 4 mm torrefied sawdust, which has very high ethylene adsorption. The mixture containing 70% vermiculite showed good results because it absorbed ethylene gas, but the results were less than expected because the fruit did not retain any noticeable freshness.

Therefore, it can be concluded that vermiculite is not as suitable as torrefied sawdust for the preparation of ethylene scavenger mixtures to maintain the freshness of apples.



a) Torrefied wood b) Vermiculite c) Torrefied wood d) Vermiculite

Fig. 3. A: Gas composition within the sealed container after 28 days of storage. The concentrations of individual gases (methanethiol, ammonia, volatile organic compounds, lower explosion limit, and ethylene) are presented using stacked bars for visualization purposes only and do not represent cumulative amounts; B: Visual changes of apple within the plastic bottle; (a) apple stored with torrefied sawdust before test; b) apple stored with vermiculite before test; c) apple stored with torrefied sawdust after 28 days; d) apple stored with vermiculite after 28 days)

Optimization of Ethylene of Mixtures Based-torrefied Sawdust

To analyze the solvent extraction process of ethylene adsorption efficiency and determine the optimal operating conditions, the response surface method (RSM) was utilized to evaluate the optimal extraction conditions for each solvent. The ranges and levels of variables and the experimental design are shown in Table 4. A quadratic model to estimate the ethylene adsorption efficiency (Y) as a function of the torrefied sawdust mix ratio (A), torrefied sawdust size (B), and ethylene absorption period (C) was applied as shown in Eq. 3. For each extraction, the objective function was to maximize the percentage of ethylene adsorption efficiency.

$$\text{Ethylene adsorption efficiency} = +83.04 + 10.97A - 2.09B + 5.65C$$

$$- 0.3927AB + 0.4450AC - 0.1309BC - 14.25A^2 - 7.92B^2 - 8.23C^2 \qquad (3)$$

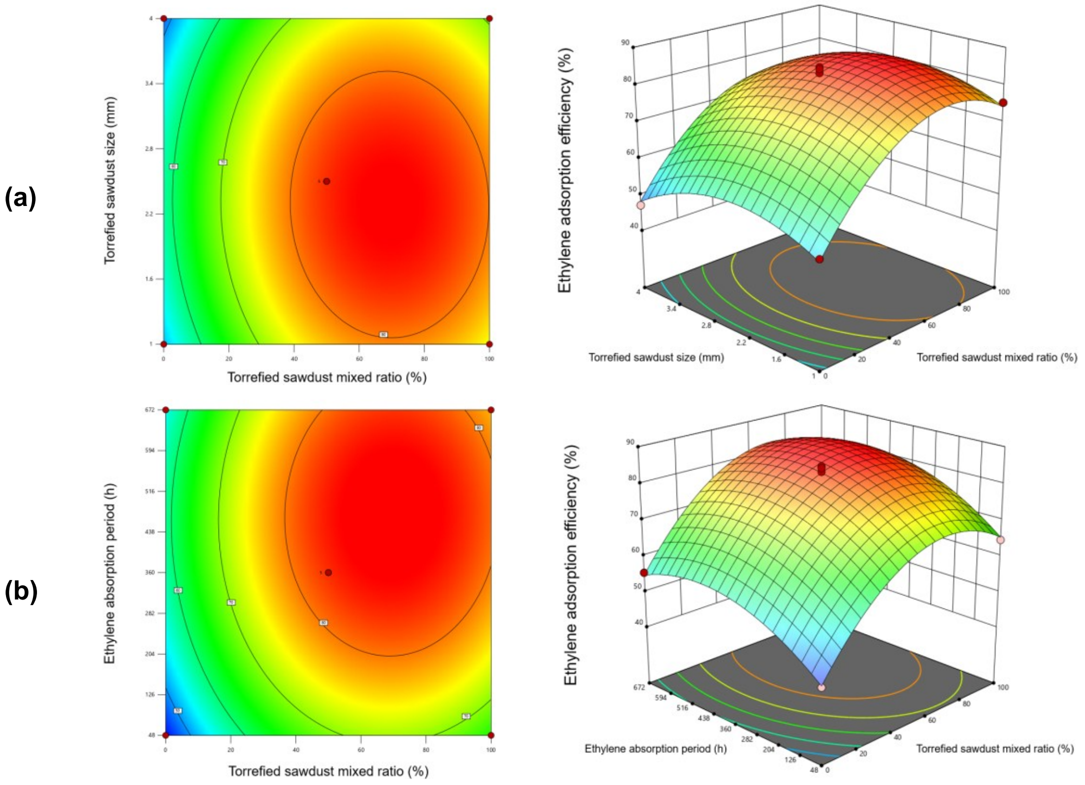
For each approximation function in Eq. 3, the analysis of variance (ANOVA) showed a model F-value of 95.68. The model R^2 for the approximation functions was 0.9919 (Table 4). This means that the model was validated and can be used within the scope of the study. The effect of the ethylene scavenger fabrication conditions design variables on the ethylene adsorption efficiency is provided in Table 4 along with the ANOVA results and model coefficients.

A response surface plot between the percentage of torrefied sawdust mix, torrefied sawdust size, and ethylene absorption period is shown in Fig. 4. The ethylene adsorption efficiency was maximized at a 66% torrefied sawdust mix ratio, 2 mm torrefied sawdust size, and 342 h ethylene absorption period, respectively. Under the optimized conditions, the ethylene adsorption efficiency was about 84% (Fig. 5).

Table 4. ANOVA of Optimization Models for Ethylene Adsorption Efficiency

Source	p-value		
Model	< 0.0001	Significant	
A	< 0.0001		
B	0.0135		
C	< 0.0001		
AB	0.6767		
AC	0.6371		
BC	0.8888		
A^2	< 0.0001		
B^2	< 0.0001		
C^2	< 0.0001		
Lack of fit	0.5066	Not significant	
$R^2 = 0.9919$			

A: Torrefied sawdust mixed ratio (%); B: Torrefied sawdust size (mm)
C: Ethylene absorption period (h)



(c)

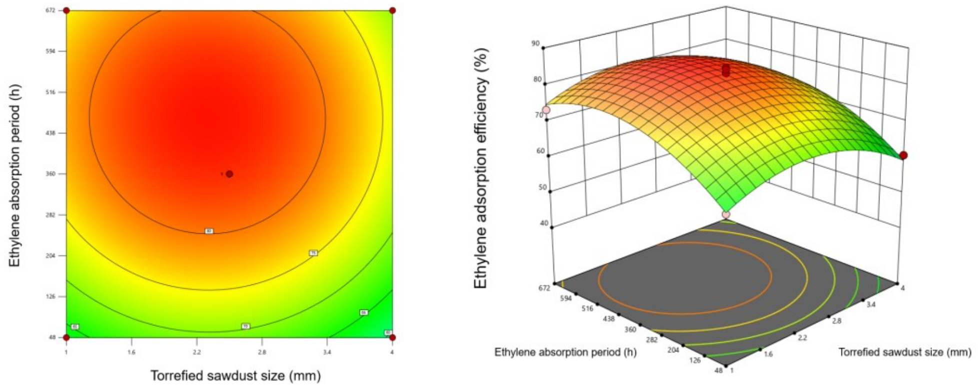


Fig. 4. Response surface plots showing the percentage of ethylene adsorption efficiency from (a) torrefied sawdust mixed ratio and torrefied sawdust size. (b) Response surface plots showing the percentage of ethylene adsorption efficiency from torrefied sawdust mixed ratio and ethylene absorption period. (c) Response surface plots showing the percentage of ethylene adsorption efficiency from torrefied sawdust size and ethylene absorption period.

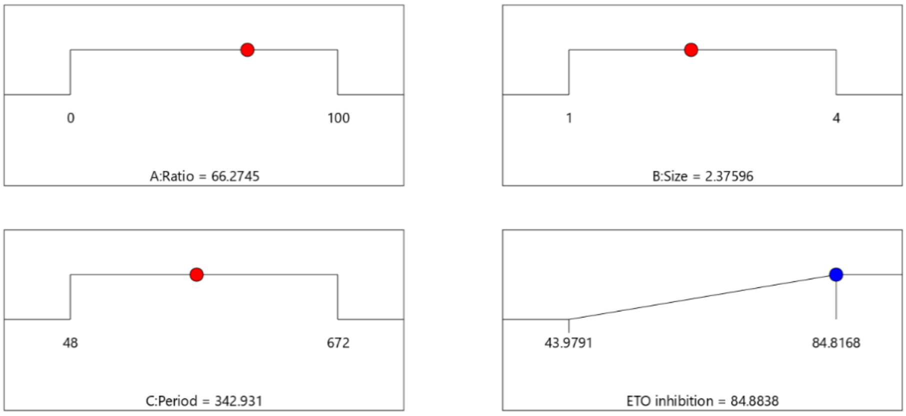


Fig. 5. Optimized scavenger preparation condition based torrefied sawdust for ethylene adsorption efficiency

Figure 4 illustrates the three-dimensional response surface plots showing the combined effects of torrefied sawdust ratio (A), particle size (B), and ethylene absorption period (C) on adsorption efficiency. As shown in Fig. 4a, increasing the torrefied sawdust ratio from 0% to approximately 66% markedly enhanced the adsorption efficiency, whereas particle sizes larger than 2 mm began to reduce performance due to decreased available surface area. In Fig. 4b, prolonged ethylene exposure time (up to 340 h) further increased the adsorption, indicating that equilibrium was reached slowly within the mixture. Figure 4c reveals that smaller particle sizes and longer absorption periods synergistically improved adsorption. Together, these surfaces highlight a clear optimum region where intermediate particle size and high sawdust content maximize adsorption efficiency.

Figure 5 presents the optimized ethylene scavenger preparation conditions generated by the Box–Behnken regression model. The software-identified optimum corresponds to a torrefied sawdust ratio of 66%, a particle size of 2 mm, and an exposure duration of approximately 342 h, yielding a predicted adsorption efficiency of ~84%. This optimized point lies within the region of maximum curvature observed in Fig. 4, confirming that the statistical model aligned well with the expected physical behavior: moderate particle size maximized surface area accessibility while maintaining sufficient pore volume, and the high sawdust proportion enhances adsorption capacity relative to vermiculite.

Previous studies have suggested that heated biomass materials may influence ethylene-related processes, yet few have quantified gas adsorption. Matan and Songsamoe (2022) reported that heated sawdust slowed mangosteen ripening, but the mechanism was not linked to direct ethylene removal. Similarly, Di Lonardo *et al*. (2013) demonstrated that biochar could reduce ethylene levels *in vitro*, although its application to fruit preservation was not evaluated.

In contrast, the present study provides a quantitative assessment of ethylene adsorption efficiency, with optimized torrefied sawdust–vermiculite mixtures achieving up to 84% adsorption, leading to more than a two-fold extension in apple shelf life. These outcomes indicate that torrefied sawdust has stronger and more measurable ethylene scavenging performance than previously reported woody materials, highlighting its potential as a practical postharvest preservation technology.

CONCLUSIONS

1. Torrefied sawdust exhibited a porous structure and proved to be an effective ethylene-adsorbing material capable of delaying apple ripening. This study represents the first systematic evaluation of torrefied sawdust–vermiculite mixtures as ethylene scavengers and their direct impact on fruit shelf-life extension.
2. The optimized mixture (comprising approximately 66% torrefied sawdust with a 2 mm particle size) achieved an ethylene adsorption efficiency of about 84%, significantly outperforming pure vermiculite.
3. Enhanced porosity and increased carbonization resulting from torrefaction contributed to the superior adsorption capacity of the mixtures, demonstrating their suitability as practical, low-cost ethylene scavengers.
4. Overall, the results confirm that torrefied sawdust–based mixtures can effectively reduce ethylene concentrations and substantially extend the postharvest shelf life of fruits, offering promising potential for commercial application in fresh-produce preservation systems.

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Data Availability

All datasets used and/or analyzed during the current study are available from the corresponding author on reasonable request.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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