Alleviating phytotoxicity of soils biosolarized with almond processing residues

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A R T I C L E   I N F O

A B S T R A C T

Biosolarization is a method for controlling soil pests and pathogens where biomass amendments are incorporated into soil and fermented under thermophilic conditions. Endogenous and fermentative organic acid biopesticides derived from the amendments can disinfest soil without the use of potentially harmful chemical fumigants. However, these biopesticides can be phytotoxic if they linger through crop establishment. This study assessed the phytotoxicity of soils amended with prominent agricultural residues – almond hulls and shells – with and without simulated biosolarization. Germination indices (GI) calculated from radish seedlings were used to measure phytotoxicity of soil amended with 0.3125 to 2.5% biomass by dry weight. Biosolarized soils were initially highly phytotoxic, and GI was negatively correlated to the concentration of succinic, acetic, and lactic acid found in treated soils. Aeration of biosolarized soil successfully improved GI and decreased organic acid concentrations. Decreasing almond residue particle size to less than 0.5 mm also aided phytotoxicity removal for certain biosolarization scenarios. This study concludes that biosolarization with low amendment rates (0.3–0.6%) would be ideal for field application, as they generated high initial organic acid accumulation (27–131 mM), but phytotoxicity could be eliminated after only one week of aeration. The results of this study will be useful for formulating land application strategies for almond residue biomass and addressing a key barrier to adoption for biosolarization as a fumigant alternative.

1. Introduction

Soil disinfestation with fumigants is often necessary to combat pest pressure from soilborne pathogens, nematodes, and weeds before planting high-value crops (Katan, 2014). Since fumigant exposure can lead to respiratory illness in

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farm workers and communities near fumigation sites (Ajwa et al., 2010; Oriel et al., 2009), there is increased interest in alternatives that disinfest soil while ensuring health and safety for people near fumigated fields (Katan, 2017). Organic soil amendments such as crop residues, manure, and compost may control soil pests via various mechanisms, including the release of bioactive chemicals and shifts in soil microbial communities (Oka, 2010). Biosolarization increases the efficacy of amendment-driving disinfestation by mulching moist, amended soil with clear tarp. This can dramatically raise soil temperatures, create oxygen stress, enrich for facultative anaerobic microbes, and promote the accumulation of pesticidal fermentation products (Momma, 2008; Simmons et al., 2016, 2014; Thoden et al., 2011). These conditions are similar to those used to produce organic acids (OA), many of which are known biopesticides, via dark fermentation (Strazzera et al., 2021). Typically, microbial activity and OA accumulation in the soil correlate to the lability of amendments used in biosolarization (Thoden et al., 2011), and immature residues have been shown to promote greater hydrolytic activity in biosolarized soils than stabilized composts (Kanaan et al., 2018).

While labile amendment degradation is crucial to soil disinfestation, unstable residues can have a phytotoxic effect (Barral and Paradelo, 2011). Field studies have observed that use of unstable soil amendments can lead to decreased crop growth compared to stabilized compost amendments; this was attributed to specific compounds produced during ammendment degradation (Bonanomi et al., 2007; Medina et al., 2015). For example, incorporating wheat straw into soil can lead to reduced crop yields due to the conversion of cellulose into acetic acid under poorly-aerated conditions (Lynch, 1977). The potential for amendments to cause phytotoxicity is a major barrier to adoption for growers and is counteractive to the pest control benefits that may be achieved using fumigation alternatives. As a result, there is a need for strategies to alleviate phytotoxicity following biosolarization. This involves balancing the amount of soil amendment required for disinfestation with an appropriate remediation period to facilitate reintroduction of oxygen to the soil and promote aerobic degradation of phytotoxic fermentation compounds. It is known that acetic acid and other OAs can be mineralized by native microbes under aerobic conditions (Malkomes, 2005). Finally, phytotoxicity may be managed by decreasing amendment particle size, which has been shown to accelerate decomposition (White and Webber, 2018), therefore reducing aeration times required ahead of planting.

The objective of this study was to gauge the inherent phytotoxicity of major almond processing waste streams and how phytotoxicity is affected by biosolarization. Due to their low cost, abundance, and co-location with fruit and vegetable production, almond hull and shell residues are of increasing interest as soil disinfestation substrates (ABC, 2020; Strauss and Kluepfel, 2015). A prior laboratory study indicated soils biosolarized with these waste streams could reduce phytoparasitic nematode populations by 84%–100%, presumably due to the observed accumulation of OAs such as acetic, formic, lactic and succinic acids (Fernández-Bayo et al., 2020). Thus, these materials show promise for use in field-scale disinfestation. In light of this valorization potential, this study sought to determine how process variables, including soil amendment rate, aeration time, and amendment particle size, could be optimized to ensure sufficient soil OA accumulation but low residual phytotoxicity. The results of this research may be used to design land application strategies for almond residue biomass and similar labile amendments and address a key challenge for growers considering biosolarization as a fumigation alternative.

2. Materials and methods

2.1. Almond residue amendments and soil preparation

Soil was obtained from Parlier, CA (36.6° N; 119.5° W; elevation 97 m), air dried, and sieved through a 2.0 mm mesh to remove any plant residues. Properties have been previously reported (Achmon et al., 2016; Fernández-Bayo et al., 2017): the moisture content was 1.15 ± 0.6 g/g wet weight, water holding capacity was 26.1 ± 0.8 g/g wet weight, volatile solids (VS) content was 0.0187 ± 0.0074 g/g dry weight, pH was 7.03 ± 0.07, and bulk density was 1.36 ± 0.12 g/cm^3. Almond hull-rich residues from the Nonpareil variety (NP) and mixed almond hull and shell residues from a pollinator variety mixture (PM) were selected as biosolarization amendments (North State Hulling Cooperative, Chico, CA). Properties of these amendments have been previously reported (Fernández-Bayo et al., 2020; Palma et al., 2020): C/N was 66 and 77, VS was 0.91 and 0.94 g/g dry weight, and pH was 4.8 and 4.7 for NP and PM biomass, respectively. Residues were ground in a laboratory blender and sieved through 0.5 mm mesh to remove large particles. To create soil mixtures containing NP or PM, soil was mixed with each residue type to achieve amendment rates of either 0.3125, 0.625, 1.25, or 2.5% on a dry weight basis. Nonamended soil was included as a control. Deionized water was added to each soil mixture stock to achieve field capacity, equivalent to 12% water content of pure soil and between 13–15% water content of amended soils. Properties of amended soil mixtures were predicted based on constituent materials (Table S1).

For experiments to evaluate the effect of residue particle size on soil and plant responses following amendment, triplicate 200 g aliquots of almond residues were ground in a laboratory blender and sieved through a series of sieves, 4.0-, 2.8-, 2.0-, 1.0-, and 0.5-mm mesh, of which three intervals were selected to represent coarse (2.8–4.0 mm), medium (1.0–2.0 mm), and fine particles (< 0.5 mm). Soil was mixed with each of the three residue size classes to achieve an amendment rate of 2.5% on a dry weight basis and then wetted to field capacity.
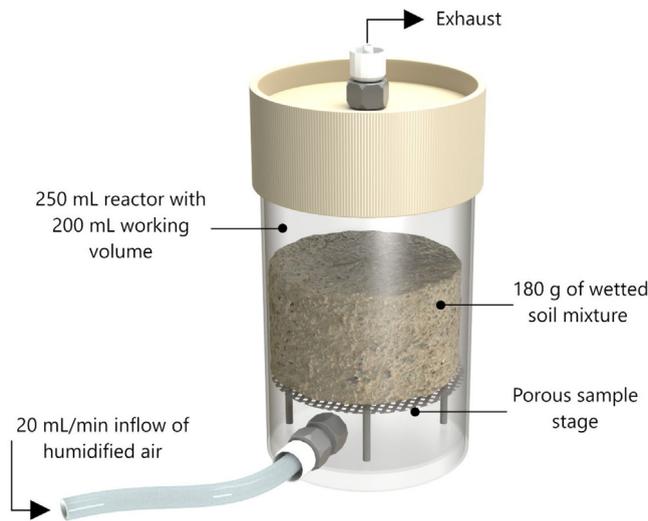
Fig. 1. Aerobic bioreactors used to remediate moist soil containing almond processing residues either immediately after amendment or following amendment and biosolarization.

2.2. Soil incubations

Anaerobic reactors were constructed by loading 240 g (wet basis) of each soil preparation into 250 mL airtight glass jars. Reactors were sealed and incubated for 8 days using an alternating temperature cycle of 12 hours at 30 °C and 12 hours at 50 °C to simulate soil biosolarization (SBS). Amended soils that underwent simulated biosolarization were designated NP SBS and PM SBS. Triplicate aliquots of 180 g of soil from the anaerobic reactors were transferred to 250 mL aerated reactors. Additionally, triplicate aliquots of freshly amended and wetted soil that was not biosolarized, coded as NP non-SBS and PM non-SBS, were used to prepare control aerated reactors. The construction of aerobic reactors has been previously described (May and VanderGheynst, 2001) (Fig. 1). Humidified air was passed through the soil at 20 mL/min for 14 to 28 days with one of three reactors removed after 7, 14 or 28 days, depending on the experiment. At each time point, soils were destructively analyzed for phytotoxicity, water content, and organic acid concentration. In total, 100 reactors were prepared to assess the effects of aeration, residue type and rate, fermentation, and particle size.

2.3. Phytotoxicity analysis

Phytotoxicity was assessed by measuring the Germination Index (GI) of radish seeds when exposed to SBS-treated and untreated soils. Forty-five mL of treated and control soils were added to Petri dishes (Falcon, NY, USA) as described by Aslam and VanderGheynst (Aslam and VanderGheynst, 2008). Dishes containing sterilized filter paper wetted with 5 mL of deionized water were prepared as negative controls. Seed incubations were performed according to previous studies (Ko et al., 2008; Mitelut and Popa, 2011): groups of 10 radish seeds (var. *Sparkler*, *Raphanus sativus*, Ferry-Morse, KY, USA) were plated in triplicate and incubated in the dark at 24 °C for 72 h. After incubation, the total number of seeds to germinate (N) was counted and the root lengths of each germinated seed were measured and averaged (L_{avg}) for each treated plate. Germination was considered positive when root lengths were at least 0.5 cm (Ko et al., 2008). These were then compared to the mean number of seeds to germinate (N_0) as well as the mean root length (L_0) of the non-amended controls to calculate GI, according to Eq. (1). Soils with a mean GI below 80% were considered phytotoxic as previously defined (Tiquia et al., 1996).

$$GI = 100\% \left(\frac{L_{avg}N}{L_0N_0} \right)$$

(1)

2.4. Organic acid analysis

The soil water content was measured as the percent weight lost after drying in a 105 °C oven for 24 h. Soil OAs were extracted from soil by combining wet soil with water at a 1:1 mass ratio in duplicate microcentrifuge tubes (15 mL, Falcon). Mixtures were vortexed for 30 s and centrifuged at 8800 g for 10 min (Eppendorf Centrifuge 5810R 15 Amp, Hamburg, Germany). The supernatant was filtered through a PVDF membrane syringe filter with a 0.2 μm pore size (Thermo Fisher Scientific Inc., San Diego, CA) into 2 mL HPLC vials (Agilent, Santa Clara, CA). Filtered soil extracts were analyzed by high-performance liquid chromatography (HPLC, model 1200, Agilent) fitted with an Aminex HPX-87H

Table 1
Main and interaction effects for simulated biosolarization (SBS treatment), type of almond biomass soil amendment (residue), amendment rate (rate), and aeration time (time) on Germination Index.

Term[a]	Estimate	STD error	Statistic	P Value
Intercept	**9.7E−01**	**9.1E−02**	**1.1E+01**	**<0.001**
SBS treatment	**−5.6E−01**	**1.2E−01**	**−4.6E+00**	**<0.001**
Residue	6.5E−02[b]	1.2E−01	5.4E−01	0.593
Rate	**−2.4E+01**	**6.1E+00**	**−3.9E+00**	**<0.001**
Time	1.2E−03	5.4E−03	2.3E−01	0.820
SBS treatment:Residue	9.9E−02[b]	1.5E−01	6.6E−01	0.508
SBS treatment:Rate	5.6E+00	7.9E+00	7.1E−01	0.477
Residue:Rate	9.1E+00[b]	7.9E+00	1.2E+00	0.249
SBS treatment:Time	**3.5E−02**	**6.9E−03**	**5.1E+00**	**<0.001**
Residue:Time	−5.5E−03[b]	6.9E−03	−7.9E−01	0.429
Rate:Time	**7.5E−01**	**3.6E−01**	**2.1E+00**	**0.039**
SBS treatment:Residue:Rate	−1.2E+01[b]	8.6E+00	−1.4E+00	0.164
SBS treatment:Residue:Time	3.0E−04[b]	6.9E−03	4.4E−02	0.965
SBS treatment:Rate:Time	**−8.8E−01**	**4.1E−01**	**−2.1E+00**	**0.035**
Residue:Rate:Time	−7.3E−02[b]	4.1E−01	−1.8E−01	0.860

[a]Bolded parameters had a significant effect on germination (P < 0.05) according to ANCOVA.
[b]The coefficient shown for residue type indicates the degree to which pollinator variety mix (PM) yielded GI values above the average value.

column (300 × 7.8 mm, Bio-Rad, Hercules, CA). The chromatography was performed with autosampler injections of 50 µL per sample, a mobile phase of 5 mM sulfuric acid in milli-Q ultrapurified water, an isocratic flow of 0.6 mL/min held at 60°C, and a total runtime of 50 mins per sample. Succinic, lactic, formic, acetic, propionic, isobutyric, and butyric acids were detected at retention times of 11.0, 11.9, 13.3, 14.4, 17.0, 19.2, and 20.9 min with a variable wavelength detector set to 210 nm (model 1260, Agilent). Standard solutions of these OAs ranging from 32.5 to 2000 mg/L were run alongside the experimental extracts and used to identify and quantify OAs in samples. Measured OA concentrations were normalized according to the moisture content of each extracted sample to yield molar concentration per unit volume of water in the original moist soil sample. Concentrations of individual OAs in each sample were summed to determine total OA concentration. Values were averaged across technical duplicates to determine the average concentration in each sample.

2.5. Statistical analysis

Germination indices, individual OA levels and total OA levels were used as response variables. Multi-way ANCOVA was performed to determine the main and interaction effects of all factors on GI and OA concentration. Depending on the experiment, this included the effects of residue type, solarization treatment, aeration time, amendment rate and/or amendment particle size. ANCOVA was initially performed with all interaction effects included; if higher-order interaction effects were not significant, they were omitted from subsequent ANCOVA. ANCOVA was followed-up by ANOVA with Tukey's honest significant difference (HSD) posthoc test to determine differences in GI between treatments at specific time points. Normality of residuals was determined using the Shapiro–Wilks test, and OA concentrations were log transformed to meet the assumptions of regression and ANOVA.

3. Results

3.1. Soil phytotoxicity

ANCOVA revealed several main and interaction effects in a multifactorial study that considered the type of almond residue used as soil organic amendment, the level of amendment in the soil, whether soil had undergone simulated biosolarization following amendment, and the duration of soil aeration following amendment or simulated biosolarization. Significant main effects were observed for simulated biosolarization treatment and amendment rate (Table 1), indicating that application of biosolarization and increasing amendment rates generally decreased GI values (Fig. 2). Although aeration time had no significant main effect, there were significant interaction effects between aeration time and amendment rate as well as aeration time and SBS treatment, indicating that GI increased with increasing aeration time for SBS treated soils, as well as soils with high amendment rates (Fig. 2, Table 1). Additionally, there was a significant 3-way interaction between SBS treatment, amendment rate, and aeration time (Table 1). This was apparent in the data as the effect of aeration time on GI depended on the combination of SBS treatment and amendment rate, with longer aeration times required to raise GI in SBS treated soils with 1.25% or 2.5% amendment rates (Fig. 2).

Residue type had no significant or interaction effects overall, but there was a significant difference in GI based on residue type in soils before aeration (i.e., the 0-day point for data in Fig. 2) according to ANOVA at this time point. The GIs of PM-amended non-SBS soils (i.e., freshly amended control soils that had not undergone simulated biosolarization)
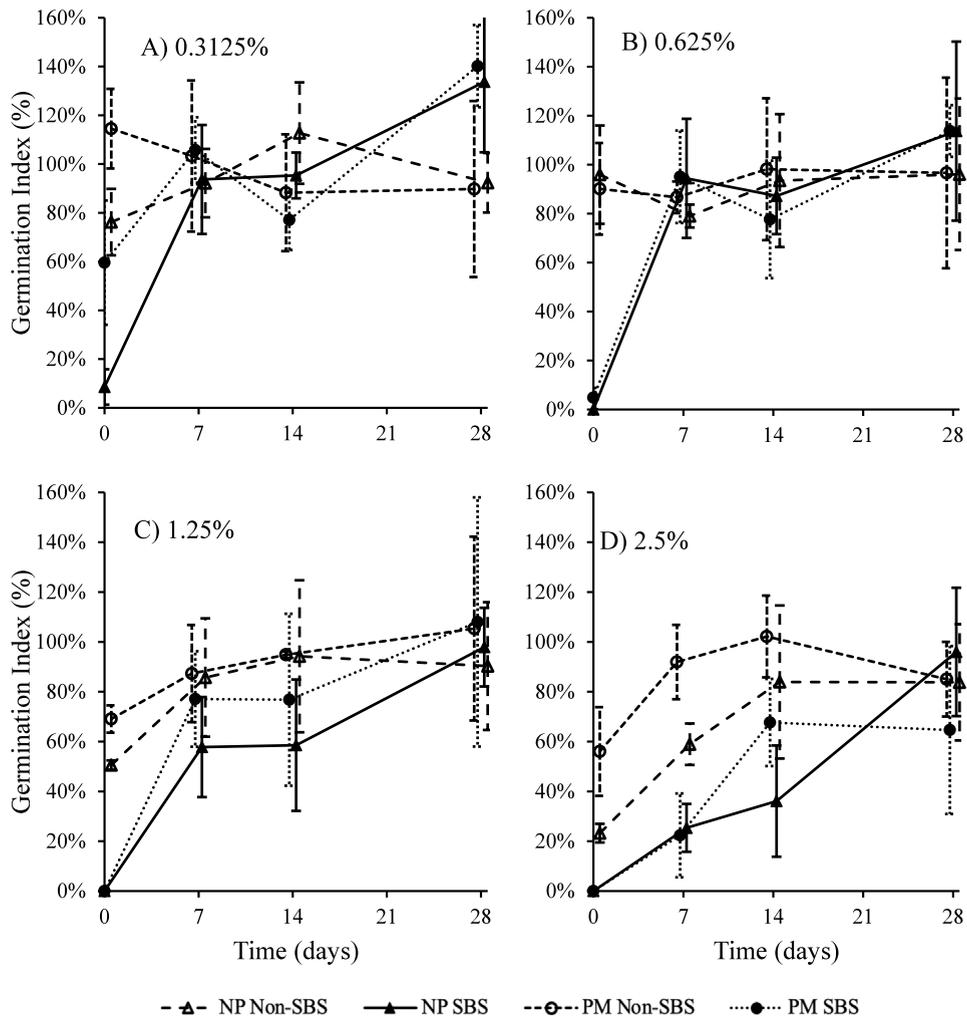
Fig. 2. Mean Germination index (GI) of soils amended with almond residues at rates of (A) 0.3125, (B) 0.625, (C) 1.25, and (D) 2.5% by dry weight. Aeration times ranged from 0 to 28 days. Lines indicate each of four soil treatment combinations: Nonpareil hull and shell (NP)-amended and pollinator hull and shell (PM)-amended soils with and without biosolarization (SBS). Data were normalized to GI values of non-amended control soils. Error bars represent one standard deviation from the mean (n = 3).

were between 9% and 45% higher than NP-amended control soils. This difference was significant at most amendment rates 0.3125, 1.25, 2.5% (P = 0.002, 0.001, 0.014, respectively), but not 0.625% (P = 0.955).

Before aeration, the GIs of non-SBS soils were between 40% and 91% higher than the SBS treatments (P < 0.001 for all rates). For non-SBS soils at this time point, a minimum of 1.25% amendment rate was needed to induce GI levels indicative of phytotoxicity, 51% and 69% for NP and PM-amended soils (Fig. 2). However, SBS induced high initial phytotoxicity at rates as low as 0.3125% for NP-amended soils (GI = 9%), and as low as 0.625% for PM-amended soils (GI = 5%). Immediately after biosolarization, extreme phytotoxicity (GI = 0%) was observed for all SBS soils with amendment rates at or above 0.625%. For soils amended with 2.5% residues, GI values in non-SBS soils were on average 52% higher than SBS soils at day 7 and 41% higher at day 14. By day 28, GI values in non-SBS and SBS soils were not significantly different.

All non-SBS soils with amendment rates at or below 1.25% had a GI of at least 80% after 7 days, and all non-SBS soils amended at 2.5% reached at least 80% after 14 days (Fig. 2). After 7 days of aeration, SBS soil with rates at or below 0.625% had a GI of at least 80%. However, 28 days of aeration was required for GI to increase to at least 80% for SBS soils with amendment rates of 1.25 and 2.5%. The exception was PM-SBS soil containing 2.5% amendment, where GI remained below 70% even after 28 days.

Residue particle size did not have a significant effect on GI (Table 2). However, there were several significant interaction effects involving residue particle size, which indicated that the effect of particle size on GI was most apparent after 14 days of aeration and for soils amended with NP residues (Table 2). Higher order interaction effects between particle size, SBS treatment and residue type showed that the extent of GI increase following aeration depended on whether SBS had
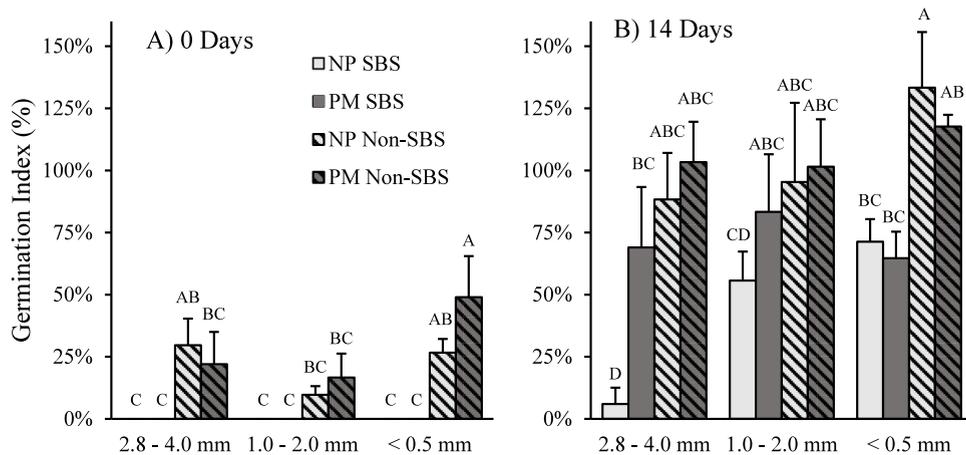
Fig. 3. Germination indices (GI) of soils amended with 2.5% almond residues (A) without aeration and (B) after 14 days of aeration. Three residue particle sizes are indicated: coarse (2.8–4.0 mm), medium (1.0–2.0 mm), and fine (< 0.05 mm) for four soil treatment combinations: Nonpareil hull and shell (NP)-amended and pollinator variety hull and shell (PM)-amended soils with and without biosolarization (SBS). Error bars represent standard deviation (n=3). Within each time point, values that do not share a letter are significantly different according to ANOVA and Tukey's HSD (P < 0.05).

Table 2
Main and interaction effects for simulated biosolarization (SBS treatment), type of almond biomass soil amendment (residue), amendment particle size (size), and aeration time (time) on Germination Index.

Term[a]	Estimate	STD error	Statistic	P Value
Intercept	**1.7E−01**	**8.6E−02**	**2.0E+00**	**0.052**
SBS treatment	−1.6E−01	1.2E−01	−1.4E+00	0.166
Residue	**2.7E−01**[b]	**1.2E−01**	**2.3E+00**	**0.024**
Size	2.7E−02	3.9E−02	7.1E−01	0.483
Time	**8.2E−02**	**8.3E−03**	**9.9E+00**	**<0.001**
SBS treatment:Residue	**−2.9E−01**[b]	**1.5E−01**	**−2.0E+00**	**0.055**
SBS treatment:Size	−3.2E−02	5.1E−02	−6.3E−01	0.531
SBS treatment:Time	**−2.2E−02**	**1.0E−02**	**−2.1E+00**	**0.041**
Residue:Size	**−1.1E−01**[b]	**5.1E−02**	**−2.1E+00**	**0.036**
Residue:Time	**−3.1E−02**[b]	**1.0E−02**	**−2.9E+00**	**0.005**
Size:Time	**−1.2E−02**	**3.6E−03**	**−3.4E+00**	**0.001**
SBS treatment:Residue:Size	**1.2E−01**[b]	**5.9E−02**	**2.0E+00**	**0.049**
SBS treatment:Residue:Time	**2.3E−02**[b]	**1.0E−02**	**2.3E+00**	**0.026**
SBS treatment:Size:Time	−3.5E−03	4.2E−03	−8.4E−01	0.407
Residue:Size:Time	**1.5E−02**[b]	**4.2E−03**	**3.7E+00**	**0.001**

[a]Bolded parameters had a significant effect on germination (P < 0.05) according to ANCOVA.
[b]The coefficient shown for residue type indicates the degree to which pollinator variety mix (PM) yielded GI values above the average value.

been applied and the combination of particle size and residue type used (Fig. 3, Table 2). Fine NP residues had significantly higher net GI increase between 0 and 14 days of aeration than coarse particles: for NP-non-SBS soils, GI of coarse residues increased from 30 to 80% after 14 days of aeration, where GI of medium residues increased from 10 to 95%, and the GI of fine residues increased from 27 to 133%. Immediately after biosolarization, all NP-SBS and PM-SBS soils had GI values of 0% regardless of particle size. For NP-SBS soils, the GI for soils with coarse residues only increased to 6% after 14 days of aeration, whereas medium particles increased to 56%, and fine particles increased to 71% (Fig. 3). PM-SBS soils showed a more consistent level of GI value increase (finishing between 65% and 83%) following 14 days of aeration regardless of amendment particle size.

3.2. Organic acid degradation

ANCOVA was only applied to SBS soils as OAs were largely non-detectable in non-SBS soils during the time course (Fig. 4). Organic acids in SBS treated soils were primarily composed of succinic, acetic, and lactic acids (Table S2). As a result, the sum of these three OAs was used to estimate total OA in soil samples. The total OAs of SBS soils increased significantly with rate and decreased significantly with time (Fig. 4, Table 3). There was no significant difference in OA levels for SBS soils amended with NP as opposed to PM residues (Table 3). For non-SBS soils, OA levels were primarily detectable in aerated soils at the initial time point (t = 0), which was measured immediately after amendment. Linear
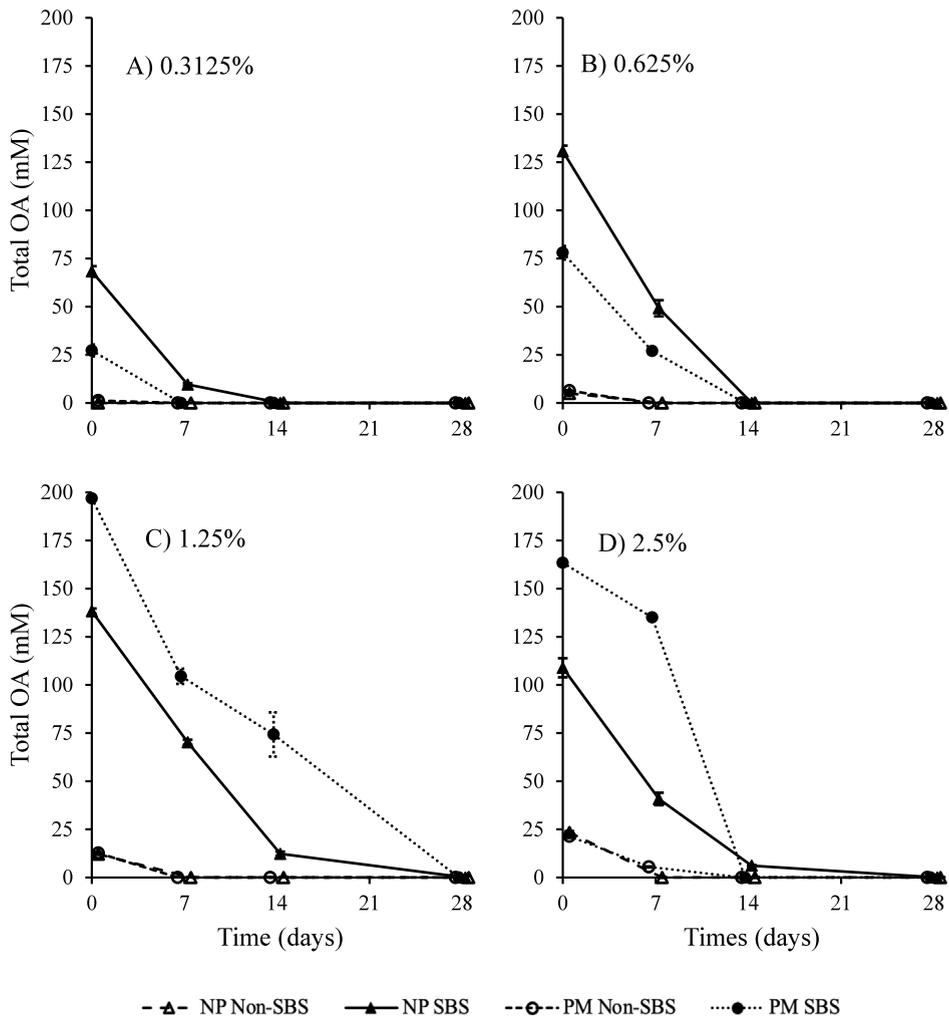
Fig. 4. Total organic acid concentrations of soils amended with almond residues at rates of (A) 0.3125, (B) 0.625, (C) 1.25, and (D) 2.5% dry weight. Aeration times ranged from 0 to 28 days. Lines indicate each of four soil treatment combinations: Nonpareil hull and shell (NP)-amended and pollinator variety hull and shell (PM)-amended soils with and without biosolarization (SBS). Error bars represent one standard deviation from the mean (n = 2).

Table 3
Main and interaction effects for type of almond biomass soil amendment (residue), amendment rate (rate), and aeration time (time) on total organic acids in biosolarized soils.

Term[a]	Estimate	STD error	Statistic	P Value
Intercept	**4.5E+00**	**1.4E−01**	**3.2E+01**	**<0.001**
Residue	1.1E−02[b]	1.7E−01	6.2E−02	0.950
Rate	**3.0E+01**	**9.2E+00**	**3.2E+00**	**0.002**
Time	**−2.7E−02**	**8.0E−03**	**−3.3E+00**	**0.001**
Residue:Time	−1.6E−04[b]	8.0E−03	−2.0E−02	0.984
Residue:Rate	−1.0E+00[b]	9.9E+00	−1.0E−01	0.918
Rate:Time	**−1.1E+00**	**4.8E−01**	**−2.3E+00**	**0.026**

[a]Bolded parameters had a significant effect on OA concentration (P < 0.05).
[b]The coefficient shown for residue type indicates the degree to which pollinator variety mix (PM) yielded total OA values above the average value.

regression applied to total OA levels in non-SBS soils at the initial time point confirmed that OA concentrations increased with increasing amendment rate (P < 0.001 for both residues).
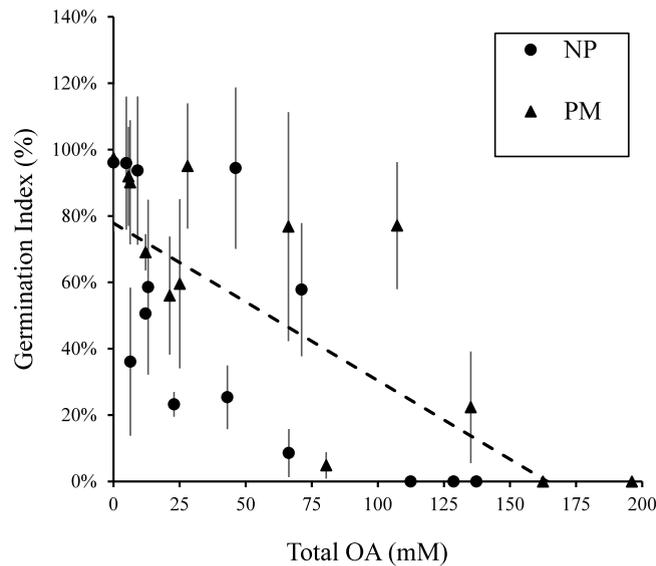
Fig. 5. Correlation between Germination index (GI) and total organic acid concentration (mM) in water within soils amended with almond residues. Points are derived from each of four soil treatment combinations, Nonpareil hull and shell-amended (NP) and pollinator variety hull and shell-amended (PM) soils with and without biosolarization, and at four aeration time points (0, 7, 14, and 28 days) and four amendment rates (0.3125%, 0.625%, 1.25%, and 2.5%, dry weight) for a total of 80 soils. Each point represents a mean of three GI replicates and 2 organic acid measurements. The solid line indicates the line of best fit from linear regression of the combined data.
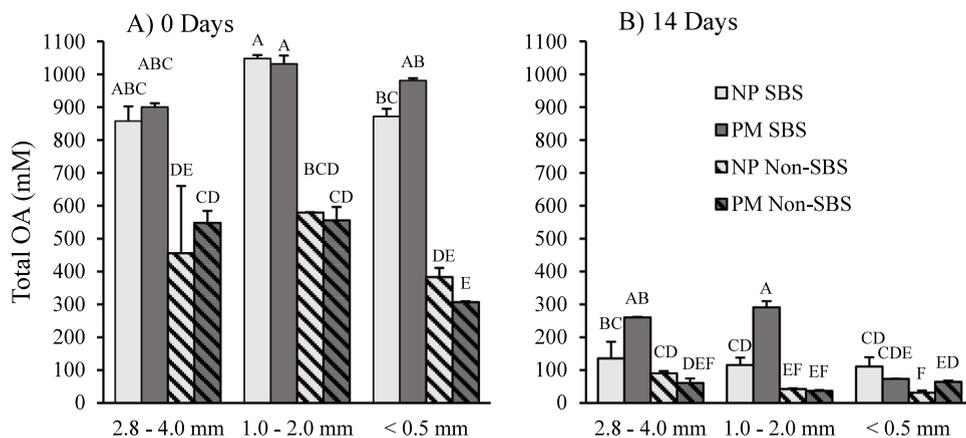

Fig. 6. Total organic acid concentration within the liquid phase of soil amended with 2.5% almond residues (A) without aeration and (B) after 14 days of aeration. Three particle sizes are indicated on the horizontal axis: coarse (2.8–4.0 mm), medium (1.0–2.0 mm), and fine (< 0.05 mm) for four soil treatment combinations: Nonpareil hull and shell (NP)-amended and pollinator hull and shell (PM)-amended soils with and without biosolarization (SBS). Error bars represent one standard deviation (n = 2). Within each time point, values that do not share a letter are significantly different according to ANOVA and Tukey's HSD (P < 0.05).

At day 0 of the aeration period, total OA levels in SBS soils were significantly higher than non-SBS soils for each amendment rate (P = 0.014 for 0.3125%, P < 0.001 for all other levels). SBS soils accumulated 27–197 mM total OAs where non-SBS soils only accumulated 1–23 mM at this time point (Fig. 4). This corresponded to soil pH values ranging neutral for 0.3125% amendment down to approximately 6 for soils with 2.5% amendment (Table S3). After 7 days of aeration, OA levels remained significantly higher in SBS compared to non-SBS soils when soil was amended with 2.5, 1.25, and 0.625% residues (P = 0.003, P < 0.001, P = 0.024) but not 0.3125% (P = 0.231). After the 7-day time point, there were no detectable OAs in non-SBS soils (Fig. 4) and soil pH returned to neutral (Table S3). Although all SBS treated soils showed eradication of OA by day 28 of aeration, SBS treated soils containing 0.625% to 2.5% amendment maintained depressed pH values ranging 6.6 to 4.7 (Table S3).

Changes in soil OA composition were detected based on the soil treatments (Table S2). Before aeration, as amendment rate increased from 0.3125 to 2.5%, succinic acid ranged from 1.2 to 22.6 mM in non-SBS soils, and 1.7–19.5 mM in SBS soils. Succinic acid was significantly more enriched in non-SBS soils compared to SBS soils (P < 0.001); this OA made up

Table 4

Main and interaction effects for simulated biosolarization (SBS treatment), type of almond biomass soil amendment (residue), amendment particle size (size), and aeration time (time) on total organic acids in biosolarized soils.

Term[a]	Estimate	STD error	Statistic	P Value
Intercept	**6.1E+00**	**2.1E−01**	**3.0E+01**	**<0.001**
SBS treatment	**7.6E−01**	**2.9E−01**	**2.6E+00**	**0.014**
Residue	−2.9E−01[b]	2.9E−01	−9.9E−01	0.329
Size	1.7E−02	9.5E−02	1.7E−01	0.863
Time	**−2.0E−01**	**2.1E−02**	**−9.8E+00**	**<0.001**
SBS treatment:Residue	3.7E−01[b]	4.1E−01	9.0E−01	0.377
SBS treatment:Size	−3.5E−02	1.4E−01	−2.6E−01	0.795
SBS treatment:Time	4.8E−02	3.0E−02	1.6E+00	0.116
Residue:Size	1.6E−01[b]	1.4E−01	1.2E+00	0.257
Residue:Time	**7.2E−02**[b]	**3.0E−02**	**2.4E+00**	**0.021**
Size:Time	**2.5E−02**	**9.6E−03**	**2.6E+00**	**0.013**
SBS treatment:Residue:Size	−1.7E−01[b]	1.9E−01	−9.0E−01	0.374
SBS treatment:Residue:Time	**−8.8E−02**[b]	**4.2E−02**	**−2.1E+00**	**0.043**
SBS treatment:Size:Time	−2.0E−02	1.4E−02	−1.4E+00	0.161
Residue:Size:Time	**−3.7E−02**[b]	**1.4E−02**	**−2.7E+00**	**0.011**
SBS treatment:Residue:Size:Time	**6.0E−02**[b]	**1.9E−02**	**3.1E+00**	**0.004**

[a]Bolded parameters had a significant effect on OA concentration ($P < 0.05$).
[b]The coefficient shown for residue type indicates the degree to which pollinator variety mix (PM) yielded total OA values below the average value.

100% of all organic acids in non-SBS soils but < 24% in SBS soils before aeration. In contrast, SBS treated soils showed significantly greater lactic acid relative abundance ($P = 0.005$). Before aeration, these levels ranged from 32 to 140 mM as the amendment rate increased from 0.3125 to 1.25% for SBS soils, accounting for 57%–83% of total OAs. Acetic acid was also detected in SBS soils, with concentrations ranging from 9 to 14 mM or 6%–34% of total OAs before aeration. After one week of aeration, 10–118 mM lactic acid was detectable in SBS soil samples, representing an average reduction of 59%; no lactic acid was detectable after 14 days of aeration in all soils save for SBS treated soils amended with 2.5% NP. Acetic acid uniquely peaked after 14 days of aeration at 12 and 72 mM in NP-SBS and PM-SBS soils utilizing an amendment rate of 1.25%, accounting for over 97% of total OAs at this time point. Overall, OAs were sparsely observed in SBS soils and the diversity of lingering OAs was reduced at 14 days of aeration (Fig. 4, Table S2).

Total OA across all amended soil samples had a significant negative effect on GI ($P < 0.001$), and this relationship was moderately strong ($r^2 = 0.55$) (Fig. 5). This relationship was stronger for NP-amended soils ($r^2 = 0.63$) than PM-amended soils ($r^2 = 0.52$). According to the 95% confidence interval for the regression, the minimum total OA concentration at which phytotoxicity is removed (GI = 80%) is predicted to be between 12.1 and 20.9 mM.

Total OA concentrations were also affected by almond residue particle size for certain soil treatments, as inidicated by significant interaction effects in the ANCOVA (Table 4). After 14 days of aeration, coarser particles (1 to 4 mm size) led to significantly greater residual OA concentrations than fine particles (< 0.5 mm size) for SBS treated soils containing PM amendment (Fig. 6). SBS treated soils containing NP amendments showed similar residual OA levels across all amendment particle sizes after aeration. While SBS treated soils containing coarse particles of PM at 2.8–4 mm and 1–2 mm generally retained greater levels of OA post-aeration compared to SBS treated soils containing NP amendment, the difference was only significant for particles in the 1–2 mm range (Fig. 6). At a particle size of < 0.5 mm, residual OA between NP and PM amendments in SBS treated soils were similar following aeration.

4. Discussion

The application of agricultural residues to soil is a common technique to improve soil properties, but can have detrimental effects on subsequent crops (Medina et al., 2015). No analytical procedure can evaluate all phytotoxic substances in amendments. Therefore, biological assays, which are sensitive to various toxins and any potential synergistic effects, are a widely accepted method of assessing phytotoxicity (Barral and Paradelo, 2011; Luo et al., 2018). The germination index (GI) is considered more sensitive to phytotoxins than relative seed germination or relative root growth alone (Emino and Warman, 2004). Sensitivity of phytotoxicity bioassays can be enhanced by directly exposing seeds to amended soils as opposed to extracts (Barral and Paradelo, 2011). Here, such direct-contact GI assays were sensitive to shifts in soil phytotoxicity caused by soil amendments, anaerobic incubation, and aerobic remediation. Though typically used to assess compost quality, these results indicate the usefulness of bioassays in other soil amendment applications, including biosolarization.

Phytotoxicity from carbon rich residues can be linked to the presence of organic acids. OAs inhibit the second phase of seed germination known as radical emergence, resulting in both decreased germination rate and stunted root growth (Luo et al., 2018). Here, soil OA concentration had a significant linear correlation to phytotoxicity. This observation is not recent; prior studies have observed that incorporating wheat straw during wet seasons was detrimental to the following

crop and attributed this effect to cellulose conversion to acetic acid under the flooded conditions (Lynch, 1977). Previous biosolarization studies similarly found that soil amendment with 5% tomato pomace and 2% mature compost led to soil acidification and decreased lettuce germination following biosolarization (Achmon et al., 2016, 2017).

In the present study, soils amended without biosolarization showed elevated levels of succinic acid and moderate initial phytotoxicity in proportion to amendment rate. Prior work identified endogenous succinic and other organic acids in almond residues that promote nematode control without fermentation (Fernández-Bayo et al., 2020). Phytotoxicity of non-fermented almond residues has been previously observed: one study found peppers grown in soilless almond hull media had 38% lower yield than the control due to potential OAs (Valverde et al., 2013), indicating that almond hull amendments require management before planting even without biosolarization. Here, organic acid concentration was further increased by anaerobic incubation. After the biosolarization treatment, total organic acids increased between 5 and 26-fold. This is in line with previous studies that found biosolarization increased OA levels by 1–7-fold compared to control soils (Fernández-Bayo et al., 2020). Here, as in previous work, this increase was the result of an accumulation of lactic acid from fermentation (Fernández-Bayo et al., 2020).

Amendment rates must be optimized to ensure effective pest control without harm to subsequent crops from residual OAs. The mechanism of biosolarization relates to the rapid formation of OAs, often in a rate-dependent matter. For instance, increasing wheat bran amendment rate from 0% to 5% resulted in total OA concentration increases of 350–1270-fold following biosolarization (Simmons et al., 2016) and 22–27-fold with almond residues (Fernández-Bayo et al., 2020). Here, total organic acid concentrations significantly increased in soils when amendment rates increased from 0.3% to 2.5%. Germination indices correlated negatively with increases in rate; previous research confirmed linear relationships between GI and concentration of phytotoxin-containing soil amendments (Emino and Warman, 2004). For biosolarized soils in the present study, even soils amended the lowest rates were phytotoxic with total OA concentrations of 27 and 68 mM for soils amended with mixed pollinator and Nonpareil varieties.

Stabilizing amendments by composting can reduce or eliminate phytotoxins before amendment (Aslam and VanderGheynst, 2008; Bonanomi et al., 2007; Medina et al., 2015), but stabilized amendments may not offer the same rapid disinfestation as unstable amendments due to depletion of labile nutrients needed for fermentative production of biopesticides in the soil (Fernández-Bayo et al., 2017). Amendment lability and residual phytotoxicity may be balanced with buffer periods to allow for aerobic remediation of phytotoxicity, which correlated to improved GI of biosolarized soils in this study. In the present study, one week of aeration was sufficient to eliminate phytotoxicity from soils biosolarized with low levels of amendment (0.3–0.6%) as well as most non-biosolarized amended soils. Up to four weeks of aeration were required to completely remove phytotoxicity from biosolarized soils with high levels of amendment (1.25–2.5%). Although total organic acid concentrations decreased with aeration time, acetic acid concentrations uniquely increased during 2 weeks of aeration for SBS soils containing 1.25% amendment. Lactic acid, the predominant OA detected in most of the SBS treated soils, is the favored product of lactic acid bacteria, enterobacteria, and clostridia under more anoxic conditions, where acetic acid is favored under microaerobic conditions (Hafner et al., 2013). Therefore, increased acetic acid concentrations during aeration may be due to mildly anaerobic conditions or microsites that remain in aerated soil due to high residual organic matter. That accumulation of acetic acid during aeration was only observed for the 1.25% amendment rate and not for lower or higher rates may suggest that particular concentration balances nutrient availability, fermentative oxygen depletion, and levels of almond residue-derived antimicrobials to temporarily favor the activity of acetogenic microorganisms (Prgomet et al., 2019). Fluctuations in potential phytotoxic compound composition during the period following biosolarization warrant further investigation with respect to their impact on specific crops.

Residue particle size selection can influence phytotoxicity by changing the rate of degradation. After noting that incorporating high levels of sugarcane residues could be detrimental to the following crop, a prior laboratory study analyzed how shredding sugarcane residues to decrease size from 2 to 0.25 mm increased residue decomposition from 61 to 91% (White and Webber, 2018). Here, residue particle size had a significant effect on phytotoxicity reduction, but only for Nonpareil residues. Soils biosolarized with 2.5% Nonpareil residues were all initially extremely phytotoxic, but the GI of soils biosolarized fine particles increased to near-healthy levels after two weeks of aeration, where soils biosolarized with coarse particles remained extremely phytotoxic. This was also correlated to organic acid concentrations, which were on average 2-fold higher in soils amended with coarse particles even after two weeks of aeration. Previous studies on wheat straw degradation found that decreasing particle size increased the degradation of the high C:N biomass by increasing contact area between soil, soil microorganisms and the residues, as well as by increasing nitrogen availability of the residues (Angers and Recous, 1997; Tarafdar et al., 2001). The effect of particle size was particularly strong in the early stages of degradation where nitrogen availability is the greatest constraint (Angers and Recous, 1997). Decreasing particle size also increases dehydrogenase activity and microbial biomass (Tarafdar et al., 2001), which may also relate to degradation kinetics.

Compositional differences between Nonpareil and pollinator varieties may relate to differences in degradation kinetics and residual phytotoxicity in response to biosolarization process variables. Pollinator variety residues generally have higher shell content and lower soluble sugar levels compared to Nonpareil variety residues (Offeman et al., 2014). Compositional analysis found pollinator variety residues had on average 36% greater fiber and 54% greater lignin, whereas Nonpareil residues had on average 41% more soluble sugar (Palma et al., 2020). Studies have shown that shells degrade slower than hulls (Valverde et al., 2013). As a result, additional work is needed to determine how much labile carbon remains in soils that have been biosolarized with almond residues from different varieties and how such carbon impacts microbial activity and OA levels during aerobic remediation of biosolarized soils.

While total organic acids correlated to phytotoxicity in the present work, it was also observed that biosolarized soils containing 1.25% or 2.5% PM or NP amendments exhibited depressed GI after 14 days of aeration, despite near complete eradication of soil OA by this time point. This mild but persistent phytotoxicity may therefore be the result of other compounds. For instance, almond residue streams contain endogenous phenolics, which may impact plant growth (Moure et al., 2007). Soil amendments may also enrich for microbes such as *Bacillus, Paenibacillus*, and *Xanthomonas* in soils, and these taxa can produce volatiles known to inhibit rice germination in vitro (Bui et al., 2019). The high nitrogen content of some amendments can result in potential ammonia toxicity and rapid fungal growth (Tan et al., 2019). The reverse can also be true, where decomposition of high C:N waste can create nitrogen deficiencies the soil (Benito et al., 2005). Future research should further explore the effects of amendment biochemical properties on soil microbial communities and soil nutrients as they relate to plant health.

5. Conclusions

Almond processing residues were found to be phytotoxic at high soil amendment rates and phytotoxicity was compounded by biosolarization. Organic acids accumulated during simulated biosolarization and correlated with phytotoxicity. The threshold at which organic acid levels were no longer phytotoxic was between 12.1 and 20.9 mM. Aeration was a successful strategy for removing phytotoxicity from soil, but up to 4 weeks of aeration was required for the most phytotoxic soil conditions. More time may be needed for remediation under field conditions due to differences in air flow, temperature, and soil moisture compared to the idealized laboratory conditions used in this study. Biosolarization using low amendment rates may offer soil pathogen control without the need for long aerobic remediation times. Decreasing residue particle size may also translate to higher degradation rates for soils amended with residues from the Nonpareil almond variety.

CRediT authorship contribution statement

Emily Shea: Conceptualization, Methodology, Validation, Formal Analysis, Investigation, Resources, Writing - original draft, Writing - review and editing, Visualization. **Zeqiao Wang:** Investigation, Validation. **Brittany Allison:** Conceptualization, Methodology, Validation, Formal analysis, Investigation, Writing - review and editing. **Christopher Simmons:** Conceptualization, Methodology, Writing - review and editing, Supervision, Project administration, Funding acquisition.

Declaration of competing interest

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

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Appendix A. Supplementary data

Supplementary material related to this article can be found online at https://doi.org/10.1016/j.eti.2021.101662.

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