Recementation of Crushed Ortstein by Leaf Extracts, Selected Organic Acids, and a Soil Amendment

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Summary. Ortstein (a soil layer cemented by organic carbon, aluminum, and iron; commonly referred to as hardpan) inhibits blueberry (*Vaccinium corymbosum*) growth. Growers have used deep tillage to break up ortstein, however, the benefits appear to have only been temporary. This study was conducted to determine if 1) crushed ortstein will recement, 2) depodzolizing species will reduce recementation, and 3) a commercially available soil amendment, Super Symbex 4X, will reduce recementation of crushed ortstein. Ortstein from Saugatuck sand (sandy, mixed, mesic Typic Durorthod) was crushed and passed through a 2-mm (0.079-inch) sieve and used in column experiments to assess recementation. Pieces of uncrushed ortstein were added to some columns to evaluate changes in cementation. Aqueous blueberry leaf extracts were added daily to columns for 1.5, 3, 6, and 12 weeks. Duplicate columns were treated with Super Symbex 4X and distilled water. Aqueous leaf extracts from bent grass (*Agrostis perennans*) and fescue (*Festuca rubra*) were added for 12 weeks. Solutions of protocathechuic acid (PCA), ρ -hydroxybenzoic acid (ρ HBA), catechol and vanillic

acid (VA) were added to crushed ortstein and allowed to stand for 5 weeks. Super Symbex 4X was added to the crushed ortstein and mixed with the organic acids at the recommended rate. Extensive recementation (96% aggregation) of crushed ortstein occurred after only 1.5 weeks of treatment with green blueberry leaf extract in the column experiments. Bent grass and fescue leaf extracts caused less and weaker recementation than blueberry. Addition of Super Symbex 4X to ortstein pieces did not produce an increase in size as did blueberry, bent grass and fescue leaf extracts. PCA, ρ HBA and VA had high levels of recementation. The water control and catechol did not show high levels of recementation. Addition of Super Symbex 4X to the organic acid and crushed ortstein decreased recementation with the strongly recementing organic acids, PCA, ρ HBA and VA. Super Symbex 4X appears to have potential to retard recementing of crushed or broken ortstein. Bent grass and fescue cover crops may not retard recementation of crushed ortstein.

Blueberries prefer acid, sandy soils with high organic matter (Lilly et al., 1975). In Michigan the most extensive soil order used for blueberry production is Spodosol. Blueberry growers in Michigan and elsewhere have observed poor plant growth when replanting older blueberry sites. Most plants reach full size and productivity, but in some parts of fields the plants are small and produce little fruit. Ortstein (hardpan) is known to reduce blueberry growth and development (Lilly et al., 1975). Hardpan is a general term that refers to layers that are cemented by different materials, whereas, ortstein refers specifically to layers that are cemented by organic carbon (C), aluminum (Al), and iron (Fe). Blueberry growers have used deep tillage before replanting to break up the ortstein. This appears to have been a temporary solution, as some Michigan growers suspect that broken ortstein recements.

Ortstein is thought to form after sufficient organo-Al and organo-Fe complexes accumulate in Bh_s and Bs horizons to cause cementation. High water tables appear to restrict downward movement of the complexes, thereby, increasing accumulations of organo-complexes and enhancing cementation. Organic acids are thought to be involved in the natural translo-

cation (podzolization) processes and may be involved in recementing broken ortstein. Protocatechuic acid was reported to have been involved in the translocation of Al and Fe in Spodosols in Michigan (Vance et al., 1986). Protocatechuic acid forms during the decomposition of plant materials and may be a degradation product of ρ HBA and VA. Protocatechuic acid, VA, ρ HBA, and catechol (Cat) have been identified as components of blueberry leaf extract (Bronick et al., 2004a). Species that promote podzolization had more of these acids than those that do not promote podzolization (Bronick et al., 2004b). Blueberries have been reported to promote podzolization in soils (Dimpleby and Gill, 1955; Miles, 1985; Mokma and Buurman, 1982). Growers may be able to limit ortstein formation by planting depodzolizing species as cover crops between blueberry rows. Bent grass and fescue were chosen for this study because they are considered depodzolizing species (Miles, 1985) and could be considered as potential cover crops between blueberry rows.

Commercially available soil amendments are promoted to reduce hardpan, but they have not been proven to decrease ortstein in blueberry fields. Super Symbex 4X (Agro-K Corp., Minneapolis) is a microbial enzyme and metabolic activator developed through the fermentation of several microbes and micronutrients. Application of Symbex resulted in a decrease in hardpan, however, the type of hardpan was not specified. The amendment stimulates natural bacterial activity that breaks up hardpan. It was suggested that the product would require 3 to 5 years to break up hardpan (L. Shaffer, personal communication).

The objectives of this study were to assess 1) if crushed ortstein would recement during treatment with leaf extract, 2) the ability of depodzolizing species to reduce cementation, and 3) the effect of a soil amendment (Super Symbex 4X) on recementation of crushed ortstein.

Materials and methods

Ortstein was collected from a blueberry field (lat. 86°08'W, long. 42°54'N) near West Olive, Mich. The soil is Saugatuck sand (sandy, mixed mesic Typic Durorthod). A trench was dug and ortstein (Bh_{sm} horizon material) extracted from the sidewalls. Some

We acknowledge Agro-K Corp. for supplying the Super Symbex 4X. Trade names are included for the benefit of the reader and does not imply endorsement of or a preference for the product by Michigan State University and does not imply its approval to the exclusion of other products that may also be suitable.

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ortstein pieces, weighing about 10 g (0.35 oz), were selected and stored in plastic bags and the remaining material was air dried, crushed and passed through a 2-mm sieve. This crushing process was more aggressive than the breaking of ortstein by tillage.

Cylindrical, plexiglass tubes 30.5 cm (12 inches) in length and 10.2 cm (4 inches) inside diameter were cut length-wise. The pieces were taped together, and then mounted vertically. The bottom was covered with cheesecloth. Crushed ortstein material was placed in the columns. Triplicate columns were treated with green or brown leaf extract for 1.5, 3, 6, and 12 weeks. References to blueberry leaf extract made without the qualifier green or brown refer to green extracts. Additional columns were treated with fescue and bent grass leaf extract for 12 weeks. Water was used as a control. The effect of topsoil was assessed by adding 2 cm (0.79 inch) of Ap horizon material, collected from the pit dug on the study site, to the top of separate columns. As the crushed ortstein was added to some columns, five air-dried, pre-weighed pieces of uncrushed ortstein were placed about 2 cm apart at different horizontally offset locations beginning at about 10 cm (3.9 inches) from the bottom of the columns. After treatment with extracts or water, the pieces were removed and evaluated for weight and strength changes.

In November and June blueberry leaves were collected from blueberry plants as well as under the plants, cleaned and stored at 5 °C (41.0 °F). Extracts from green blueberry leaves were thought to represent the result of rainwater flowing over green leaves and those from brown leaves represented rainwater that flowed over senesced brown leaves. The leaf extracts likely had higher concentrations of organic materials than rainwater that had flowed over leaves. Leaves and grass clippings were air dried, placed in plastic bags, and crushed by hand. Fescue, bent grass and brown blueberry leaf extracts were prepared weekly. Green blueberry leaf extracts were prepared daily to minimize changes in composition during storage of the extracts. Crushed leaves were placed in distilled water at 100 g·L⁻¹ (13.35 oz/gal), held for 24 h at 20 °C (68.0 °F), then filtered through a Buchner funnel. A volume of 20 mL (0.68 fl oz) of extract or water was gently poured over the column

surface daily during a one-minute interval attempting to evenly distribute the liquid. This amount corresponds to approximately twice the mean daily precipitation in the region. Super Symbex 4X (hereafter referred to as Symbex) solution was added at 45-day intervals using the recommended rate of 2.3 L·ha⁻¹ (1 qt/acre).

At the conclusion of the treatments, the columns were allowed to air dry and then were separated in 3-cm (1.2-inch) increments using a spatula. Materials from each segment were passed through a 2-mm sieve and the cemented material remaining on the sieve was weighed. We measured only the upper 12 cm (4.7 inches) of each column, since lower portions were thought to be at or near saturation before leachate would drip from the column.

Air-dried ortstein pieces were weighed for comparison to original weights. Cemented materials were assessed for their structural tensile strength (in 3-cm increments) using a polar tensile strength press as modified (A.J.M. Smucker, personal communication) from Dexter and Kroesbergen (1985). Cemented pieces were placed in a mist chamber for 24 h, set in plastic bags for 24 h and then gently broken along natural breaks and allowed to air-dry. Representative cemented pieces were randomly selected for tensile strength from the 6.3- to

9.5-mm-diameter (1/4- to 3/8-inch) fraction. About 10 cemented pieces were tested for each replicate.

The tensile strength press consisted of two parallel plates. One plate was placed on a scale and the second, a moveable plate, was computer-controlled. A cemented piece was placed between the two plates. The moveable plate incrementally moved downward, exerting pressure on the cemented piece and scale. The pressure was measured on the scale and the computer recorded the pressure. The maximum force required to deform or break a cemented piece was recorded. For pieces that gradually deformed, the maximum stress required to initiate deformation was recorded. For pieces that broke abruptly, the appropriate peak strength was recorded. The crushed piece was weighed, dried at 105 °C (221.0 °F) for 24 h and weighed again. Only the actual force required to break the pieces was used, owing to size changes in the original pieces. Additional material on some of the pieces formed hybrid structural units with varied tensile strengths. The force values were compared to those of the original ortstein.

Organic acid solutions [0.05 g·L⁻¹ (0.0067 oz/gal) distilled water] and organic acids and Symbex were added to a 5-g (0.18-oz) sample of crushed ortstein in 200-mL conical flasks. Water and water with Symbex and crushed

Fig. 1. Recementation [>2 mm (0.079 inch)] in the top layer [0 to 3 cm (1.2 inch)] for crushed ortstein treated with green or brown blueberry leaf extracts. Error bars represent one standard deviation ($n = 3$).

Fig. 2. Recementation [$>2\text{mm}$ (0.079 inch)] for crushed ortstein with depth after treatment for 12 weeks with blueberry (green leaves), bent grass, or fescue leaf extracts or water. Error bars represent one standard deviation ($n = 3$); $2.54\text{ cm} = 1.0\text{ inch}$.

Fig. 3. Tensile strength of uncrushed ortstein and recemented materials in the top layer [0 to 3 cm (0.079 inch)] of columns of crushed ortstein treated with blueberry (green leaves), fescue or bent grass leaf extracts. Letters indicate significant differences between treatments using least squared means ($P = 0.05$); $1\text{ MPa} = 9.9\text{ atmospheres}$.

ortstein were used as controls. The mixtures were allowed to stand for 5 weeks at room temperature. At the end of the treatment, the supernatant solutions were decanted and the crushed ortstein materials were air dried and then passed through a 2-mm sieve. The

air-dry weight of the loose material and aggregated material (remaining on the 2-mm sieve) were compared.

Data were analyzed using mixed model procedure (Proc mixed) with repeated statement in SAS statistical software (SAS Institute, 1989). The

logarithmic transformation was applied to weight and strength data to provide normality assumption for analysis. Differences between treatments were determined by using least squared means (LSMEANS) differences in the ($P < 0.05$) significance level with the Tukey-Kramer adjustment.

Results and discussion

RECEMENTATION. More than 95% of the top layer (0 to 3 cm) of columns treated with green blueberry leaf extract was recemented at the first sampling time, 1.5 weeks (Fig. 1). Brown leaf extract treatments resulted in less recementation, 40%, in the top layer at the first sampling. This is likely the result of green blueberry leaf extract having higher concentrations of organic acids than brown leaf extracts. At the end of 12 weeks, about 100% of the crushed ortstein in the top layer had recemented. While separating the columns into 3-cm segments with a spatula, some sand grains were broken from the recemented material. Therefore, the percent recementation reported is less than actual.

Blueberry, bent grass or fescue leaf extracts resulted in greater than 80% recementation in the top layer after 12 weeks (Fig. 2). Recementation decreased with depth, however, there was no significant difference ($P > 0.05$) between recementation in the 6- to 9-cm (2.4- to 3.5-inch) and the 9- to 12-cm (3.5- to 4.7-inch) layer. Less than 25% of the upper two layers of the water treated columns was cemented. The addition of Ap horizon material did not significantly ($P > 0.05$) affect recementation in the top layer (0 to 3 cm) by green blueberry leaf extract (88 and 90%) and fescue extract (79% and 85%). The hypothesis of some Michigan blueberry growers that broken ortstein has recemented is likely correct since deep tillage likely does not break the ortstein as completely as the crushing process.

TENSILE STRENGTH OF AGGREGATES. Tensile strength of aggregates in the top layer was assessed for each extract treatment (Fig. 3). Tensile strength of recemented material from the blueberry extract or bent grass treatments were significantly ($P < 0.05$) greater than that of the fescue extract treatments. Tensile strength of the recemented materials from the blueberry extract treatments was not significantly ($P > 0.05$) greater than that of the uncrushed ortstein.

Fig. 4. Weight changes in uncrushed ortstein pieces after treatment with blueberry (green leaves), fescue, and bent grass leaf extracts and Symbex. Letters indicate significant differences between treatments using least squared means ($P = 0.05$).

Fig. 5. Force required to crush ortstein pieces following treatment. Letters indicate significant differences between treatments using least squared means ($P = 0.05$); 1 g = 0.04 oz.

Root pressure has been recorded at less than 1 Mpa (9.9 atmospheres) for pea (*Pisum sativum*) plants (Clark et al., 1999). This was exceeded by the aggregates from all three-extract treatments. The tensile strength of aggregates from the top layer exceeded 1 Mpa, including the 1.5-week blueberry treatment. This does not provide conclusive evidence that recemented materials cannot be penetrated by roots, however, it does suggest that the recemented materials are likely to have a negative effect on root growth

and, therefore, productivity.

UNCRUNSED ORTSTEIN PIECES. Weight changes of ortstein pieces were determined based on air-dry weights before and after treatment. Pieces treated with blueberry or fescue extracts had significant ($P = 0.05$) increases in weight (215 and 118%, respectively), whereas, those treated with bent grass extract, Symbex or water had no significant ($P > 0.05$) increase in weight (Fig. 4). Ortstein pieces treated with Symbex or water weighed less after treatment than be-

fore treatment. Bent grass and Symbex appeared to retard recementation of crushed ortstein compared with fescue. The addition of Ap horizon material to columns did not significantly affect the weight of the uncrushed ortstein pieces treated with blueberry leaf extract (288% and 215%) or fescue extracts (118% and 80%). The increase in weight, and therefore size, as a result of addition of blueberry extract suggests that pieces of ortstein resulting from deep tillage in blueberry fields would also grow in size and may in fact be welded to other pieces.

All treated pieces were weaker than the uncrushed ortstein (Fig. 5), however there was no significant difference ($P < 0.05$) between the strength of bent grass treated pieces and uncrushed ortstein. Ortstein pieces from columns treated with bent grass extracts were stronger ($P < 0.05$) than those treated with blueberry extract. Pieces that received both blueberry extracts and Symbex were not weaker than those that received only blueberry extracts. Bent grass extracts inhibited size increase, although after 12 weeks they did not reduce the strength of ortstein pieces. Fescue extracts altered the pieces of ortstein, forming hybrid units with a strong core and more friable outer zone with reduced strength. It is not clear if bent grass and fescue can reduce the strength of existing ortstein and may be good cover crops to use to reduce recementing of broken ortstein. Pieces treated with fescue or Symbex were about the same strength as those treated with water. The addition of Ap material did not significantly ($P < 0.05$) affect the strength of the pieces treated with blueberry leaf extract [1072 and 1007 g (37.8 and 35.5 oz)] and fescue extract [737 and 742 g (26.0 and 26.2 oz)]. These laboratory results suggest that water and Symbex may help prevent recementing of ortstein broken by deep tillage for blueberry production.

RECEMENTATION BY SELECTED ORGANIC ACIDS. Organic acids are thought to be involved in the movement of Al and Fe in soil to cause cementing of sand. Therefore, we chose three organic acids (PCA, VA, and pHBA) that are considered strong complexing materials and one (catechol) that is considered a weak complexing material. The PCA, VA, and pHBA treatments had larger ($P < 0.05$) amounts of recementation than catechol and

Fig. 6. Recementation of crushed ortstein for selected organic acids, protocatechuic acid (PCA), vanillic acid (VA), ρ -hydrobenzoic acid (pHBA) and catechol (Cat) with and without Symbex. Letters indicate significant differences between treatments using least squared means ($P = 0.05$).

water treatments (Fig. 6). The amounts measured are likely less than actual because much material adhered to the container and some aggregates broke up during transfer. Recementation is thought to result from organic acids complexing Al exposed on the sand grains and transporting it to contact points of adjacent sand grains. PCA had the greatest recementation. This is consistent with findings that PCA appears to play a role in podzolization (Vance et al., 1986). Symbex appeared to weaken existing ortstein and prevent cementing of broken ortstein, therefore, Symbex was added to the organic acids. Organic acids, PCA and VA, that had high recementation alone, had less ($P < 0.05$) recementation when Symbex was added; pHBA also had less ($P > 0.05$) recementation when Symbex was added. Water alone and Symbex appeared to prevent the formation of Al complexes.

Conclusion

Recementation of crushed ortstein by aqueous blueberry leaf extracts occurred in less than 1.5 weeks. Blueberry and bent grass leaf extracts had greater ability to recement crushed ortstein and their recemented materials were stronger than fescue leaf extracts. Ort-

stein pieces increased in weight after receiving blueberry and fescue leaf extracts, but not those receiving bent grass leaf extracts or Symbex. Symbex reduced the amount of recementation of crushed ortstein when used with blueberry extract. PCA, pHBA, and VA produced more recementation than catechol and water. Addition of Ap material did not affect recementation or weight or strength of uncrushed ortstein pieces. Symbex was able to decrease recementation in strongly cementing organic acids. Based on this laboratory study, blueberry leaf extracts likely cause recementing of broken ortstein. It is probable that the recemented ortstein negatively impacts blueberry root growth. Deep tillage alone is not likely a permanent solution to the problem caused by the presence of ortstein in soils used for blueberry production. It is not clear whether depodzolizing species, such as bent grass and fescue, will successfully prevent broken ortstein from recementing and/or weaken existing ortstein, and whether they will be good cover crops between blueberry rows. Fescue appears to be a better choice than bent grass. Super Symbex 4X appears to be useful in retarding the recementation of disturbed ortstein in

blueberry fields following deep tillage. Water alone also appears to retard recementation, therefore, irrigation after breaking the ortstein layer and before planting blueberries may be beneficial. After planting blueberries it is likely that water added by above ground irrigation will contain organic acids from the blueberry plants. Drip irrigation may be a more successful long-term solution because the water does not come in contact with the canopy or decomposing leaves. The results of this study should be tested in the field.

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