

AGRONOMY AND SOILS

Phosphorus Recovered From Swine Wastewater as a Fertilizer for Cotton Grown With Conservation Tillage

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ABSTRACT

Current technologies for recycling P from animal waste through precipitation result in non-conventional fertilizer products. The objective of this research was to evaluate the use of surface broadcasting recovered calcium phosphate as a P fertilizer source for cotton (*Gossypium hirsutum* L.) when grown after winter rye (*Secale cereale* L.) biomass crop using conservation tillage. Treatments in this two-year field study were fertilizer material [no P fertilizer, recovered P with particle size of 0.5 – 1.0 mm, recovered P with particle size of 2.0 – 4.0 mm, and diammonium phosphate (DAP)]. Fertilizer application rate was 168 kg P₂O₅ ha⁻¹. Plant available P (Mehlich 1) and soil water P in the surface 10 cm were measured monthly. Crop biomass and P uptake and cotton seed P were determined. The ranking among treatments both years for plant available soil P was recovered P in 0.5 – 1.0 mm particles > recovered P in 2.0 – 4.0 mm particles = DAP > control. For soil water P, DAP was consistently higher than the control, but the two particle sizes of recovered P were similar to the control. No differences occurred in rye growth or P uptake among the two recovered P treatments and the control either year. The DAP increased rye biomass and P uptake early in the season in the second year of the study, but not in the first. Treatments did not differ for cotton P uptake or yield. When used as a fertilizer, recovered calcium phosphate should be applied in small particles.

Southeastern cotton producing areas are also areas of intense livestock production, particularly poultry and swine. Interest has recently

increased in recovering P from animal manure because of environmental concerns for ecosystems in areas with a high density of animals (Kaiser et al., 2009; Schroeder et al., 2004). In addition to this, growing global demand for food and fiber will require more judicious use of world P resources (Cordell et al., 2009; Keyser et al., 2010). An animal manure treatment system has recently been developed that recovers P from liquid manure as calcium phosphate (Vanotti et al., 2003). Although this manure treatment byproduct contains a lower percentage of P in a water soluble form than currently used commercial P fertilizers, the plant available P fraction is as high as in commercial fertilizers (Bauer et al., 2007).

There has been a considerable amount of research on application of non-mobile nutrients, especially P, in conservation tillage systems because of concerns that the lack of mixing of fertilizer with the soil could result in crop nutrient deficiency. It is well-established that in conservation tillage systems P accumulates near the soil surface (Dick, 1983; Edwards et al., 1992; Karlen et al., 1989; Lal, 1976; Tripplett and van Doren, 1969). Even though stratification occurs with conservation tillage, it generally does not reduce crop yield or P uptake (Howard et al., 1999; Touchton et al., 1982) unless soil surface conditions are dry for long periods and soil lower in the profile has low nutrient availability (Mallarino et al., 1999). In fact, occasional tillage of fields managed with no-tillage to incorporate surface applied nutrients does not appear to increase crop yields (Quincke et al., 2007) or P uptake at harvest (Garcia et al., 2007).

Previous research on P fertilizer management with conservation tillage included commercial fertilizers and animal manure; both P sources contain a substantial proportion of the P content as water soluble (Chien et al., 2011; Sistani et al., 2003). Total solubility of P fertilizer materials is calculated as the sum of the water-soluble and citrate-soluble portions of the material (AOAC, 2000). The calcium phosphate recovered from swine wastewater (Vanotti et al., 2003) has a total P solubility of more than 99%;

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98% citrate soluble but only <1% water soluble (Bauer et al., 2007). Because of its high citrate solubility, calcium phosphate recovered from swine wastewater appears to be a suitable fertilizer for supplying P to plants when incorporated into the soil. However, its utility as a fertilizer to rapidly supply P appears to be particle size dependant (Bauer et al., 2007), as has also been reported for water soluble P fertilizer materials (Sander and Eghball, 1988).

Calcium phosphate recovered from animal waste could be further processed into more water-soluble fertilizer materials. A more efficient use of this material would be to apply it directly to fields requiring P fertilizer. Because calcium phosphate from animal waste treatment has lower water solubility, it may require altered management to optimize its use as fertilizer in conservation tillage systems. The objective of this research was to evaluate the potential of manure-recovered calcium phosphate as a P fertilizer source for a biomass rye-cotton double crop system using surface broadcasting and conservation tillage.

MATERIALS AND METHODS

Recovery and Production of Phosphorus Materials. The recovered phosphorus was provided by a full-scale liquid manure treatment facility that treats waste from a 4360-head finisher swine production unit. In the treatment process, soluble P in the wastewater is recovered by increasing the pH of the wastewater with controlled amounts of hydrated lime to precipitate the P as calcium phosphate (Vanotti et al., 2007). The calcium phosphate is relatively concentrated in P (114 g kg⁻¹, Bauer et al., 2007). Following the precipitation process, the calcium phosphate precipitate is dewatered using filter bags and air-dried into a caked material (Vanotti et al., 2007).

The recovered calcium phosphate was tested for process ability into commercial fertilizer-sized granules by the International Fertilizer Development Corporation (IFDC) in Mussel Shoals, Alabama. After initial testing, the IFDC processed 249 kg of the material into fertilizer-sized granules. Two sizes were produced; particles that passed through a 1.0 mm sieve but were retained on a 0.5 mm sieve, and particles that passed through a 4.0 mm sieve but were retained on a 2.0 mm sieve.

Field Study. A field study was conducted at the Clemson University Pee Dee Research and

Education Center near Florence, SC in 2008-2009 and 2009-2010. Routine soil testing in 2006 of the surface 15 cm of the soil in the field used in this study indicated 'medium' soil test (Mehlich 1) P. In 2007 and 2008, the field was cropped in corn (*Zea mays* L.) with no added P fertilizer. Soil fertility parameters of the surface 10 cm of the specific areas of the field at the time of rye planting in the fall of each year are shown in Table 1. Areas of the field used for this study had soil types of Norfolk loamy sand in 2008-2009 and Bonneau sand in 2009-2010.

Table 1. Soil fertility characteristics of the surface 10-cm of the soil in November of 2008 and 2009.

Year	pH	P	CEC	K	Ca	Mg
		mg kg ⁻¹	-----	cmol kg ⁻¹	-----	
2008-2009	5.8	20.1	3.73	0.12	1.35	0.32
2009-2010	5.6	16.0	2.82	0.08	0.95	0.22

The study had four treatments: recovered calcium phosphate in particles sized 0.5 – 1.0 mm, recovered calcium phosphate in particles sized 2.0 - 4.0 mm, commercial diammonium phosphate (DAP), and a control that did not receive P fertilizer. The recovered calcium phosphate and diammonium phosphate were applied at a rate of 168 kg P₂O₅ ha⁻¹ (74 kg P ha⁻¹) in plots that were 3.85 m wide and 10.64 m long. Treatments were arranged in a randomized complete block experimental design with four replicates in each year.

Rye (cv. Wren's Abrussi) was planted on 5 November 2008 and 16 November 2009 with a John Deere model 750 grain drill. Seeding rate was 112 kg seed ha⁻¹. One (2008) or two (2009) days later, the phosphorus treatments were applied to the plots by hand-spreading the fertilizer materials to the soil surface in each plot. To increase the uniformity of the application, approximately one-half of the material was applied walking in one direction and the other half was applied walking perpendicular to the first pass. After the P application, N was applied as Ca(NO₃)₂ to all plots with a drop spreader. The control and the two recovered P treatments received 67 kg N ha⁻¹ in this application. Since the DAP application provided approximately 30 kg N ha⁻¹, the plots receiving DAP received 37 kg N ha⁻¹ as Ca(NO₃)₂.

Each year, phosphate released into the soil solution was measured using polypropylene fiber mesh membranes covered with anion exchange resins.

Sheets (10 cm X 10 cm) of anionically-charged membrane material (GE Ionics, Cambridge, MA) were activated with dilute NaHCO_3 and attached to slightly larger pieces of styrofoam. Immediately prior to the phosphorus application, these anionic membranes were buried vertically into the soil so that the top of the membrane was level with the soil surface. Two sheets of anionic membrane were randomly placed in each plot. Anion exchange membranes were collected and replaced at monthly intervals from December through May of both years. After the membranes were collected, they were placed in a sealed plastic bag and transported under ice to the laboratory. Phosphate sorbed to each membrane was displaced using 1 M NaCl solution and P was quantified colorimetrically using the ascorbic acid method with a Technicon autoanalyzer (APHA, 1998).

Plots were sampled monthly from November through September for available P in the surface 10 cm of soil each year. Eight 2.5 cm diameter cores were collected from each plot and air-dried. The samples were then sent to the Clemson University Extension Agricultural Service Laboratory for P (Mehlich 1) analysis.

Rye biomass and P content was measured on plant samples collected on 19 March, 9 April, and 6 May in 2009 and 18 March, 14 April, and 3 May in 2010. Samples were collected by clipping all plants at the soil surface in a 0.38 m² area from the middle of the plots. Plant samples were dried at 65°C for three days, weighed, and ground. After the last sampling date each year, the rye was cut with a disc mower, allowed to dry in the field, and then raked and baled to remove the biomass from the plot area.

Tillage was limited to the use of an in-row subsoiler just prior to planting cotton. Cotton (cv. DPL 0949B2RF) was planted on 13 May 2009 and on 20 May 2010 with a Case-IH model 900 four-row planter equipped with wavy coulters. Rows were spaced 0.96 m apart. Seeding rate was approximately 11 seeds per m of row. Aldicarb (0.8 kg a.i. ha⁻¹) was applied in-furrow during planting for control of thrips (*Frankliniella* sp.). Foliar insecticides were applied as needed to control other insect pests {primarily stink bugs [*Acrosternum hilare* (Say), *Nezara viridula* (L.), and *Euschistus servus* (Say)]}. Weeds were controlled using a combination of herbicides (at recommended rates and timings) and hand weeding as necessary.

Cotton biomass samples were collected during the early flowering period on 26 June 2009 and 14 July in 2010. All plants in 0.5 m of row were clipped at the soil surface, dried, weighed, and ground as described for the rye biomass samples. Two interior rows of cotton were harvested in each plot with a spindle picker equipped with a weigh basket. Cotton was harvested on 6 November 2009 and 21 October 2010. Samples of the cotton were collected after weighing and saw-ginned to determine lint percentage. After ginning, the seed cotton from the sample was ground for determination of P content.

Phosphorus content of the rye biomass, cotton biomass, and cotton seed was determined. Duplicate samples (0.15 g each) were digested in 50 mL digestion tubes using an Environmental Express Auto-block, Model SC860 and the following procedure: 5 mL concentrated HNO_3 were added and allowed to sit for 24 hours at room temperature. The block was heated to 70°C (equivalent to 60°C sample temperature) and maintained at this temperature for 30 minutes. The samples were then allowed to cool for 35 minutes prior to adding 2 mL of Milli-Q H_2O and 5 mL of concentrated H_2O_2 . Following these additions, the block was heated to 112.5°C (equivalent to 100°C sample temperature) for 2.5 hours. The samples were then allowed to cool for 30 minutes and diluted to 50 mL volume with Milli-Q H_2O resulting in a 6% HNO_3 solution. These solutions were filtered through 0.2 μm PTFE (teflon) filters and analyzed for P with a Varian Vista-Pro simultaneous ICP-OES (Inductively Coupled Plasma-Optical Emission Spectrophotometer).

All data were subjected to analysis of variance. Analysis was conducted by year using the GLIMMIX procedure in SAS to avoid confounding soil type with weather conditions over the two years. Means for the treatments were separated using the PDIF mean separation technique.

RESULTS AND DISCUSSION

Fertilizer Processing Results. The recovered calcium phosphate was relatively easy to process into fertilizer-sized particles. Tests by the IFDC suggested compaction followed by granulation and sieving was a feasible procedure for processing the material into fertilizer granules. Importantly, the material compacted best at a moisture

content of about 20% (w/w). Thus, air-drying of the material after it is precipitated from the wastewater will likely be sufficient for on-farm processing of the material before it is delivered to a fertilizer processing facility. The process of compaction followed by granulation was over 80% efficient in providing the particle sizes used in the field study.

Soil Response. In both years of this field study, the difference in water solubility between DAP and the two particle sizes of recovered P was clearly demonstrated by the amounts of P captured on the anionic exchange membranes. At no sampling time did the two recovered P fertilizer particle size treatments differ from each other or from the control (Table 2). Phosphorus captured on the membranes in the plots where DAP was applied, on the other hand, was higher than the control at every sampling time. For DAP, most of the P captured on the membranes occurred during the first month after fertilizer application (Table 2). This was likely due to the substantial rain that occurred during that period in both years. Total rainfall was 119 mm during the first 30 days after fertilizer application in 2008 and there were a total of five rain events of greater than 1 mm. In 2009, initial 30-day total rainfall was 104 mm and there were ten rain events of greater than 1 mm. In both years, there were two rain events during that period that totaled at least 25 mm of precipitation.

Table 2. Effect of phosphorus fertilizer source on P captured by ion exchange resins in the surface 10 cm of soil during 2008-2009 and 2009-2010.

Year	Month	Fertilizer Source			
		Control	0.5 – 1 mm	2 – 4 mm	DAP
-----µg cm ⁻² -----					
2008-2009	December	3.8b	3.9b	6.0b	172.2a
	January	0.2b	0.0b	0.5b	12.1a
	February	0.7b	1.0b	0.7b	8.1a
	March	0.0b	0.7b	1.4b	5.8a
	April ^z	-	-	-	-
	May	0.0b	1.2b	0.0b	3.4a
2009-2010	December	1.4b	2.8b	1.9b	254.4a
	January	1.1b	2.4b	3.6b	10.5a
	February	0.3b	1.4b	1.4b	4.7a
	March	0.4b	1.0b	1.5ab	3.4a
	April	0.6b	0.8ab	1.7ab	2.9a
	May	0.0b	0.1b	0.2b	0.9a

^z Resin sheets were damaged so data could not be collected for April of 2009.

In contrast to the anion exchange membrane data, average plant available P in the surface 10 cm of soil during the rye and cotton growing seasons each year was greatest for the 0.5 – 1.0 mm sized particles of the recovered P treatment (Table 3). Soil in plots receiving recovered P in particle sizes of 2.0 – 4.0 mm was similar in plant available P to plots receiving DAP. The control treatment had the lowest plant available P of the four treatments. A month by treatment interaction occurred both years for plant available P. Plant available P in the control treatment was consistent throughout the experimental period each year. The magnitude of differences from the control for the other three treatments and the ranking among the other three treatments differed among months (Figure 1).

Table 3. Plant available (Mehlich 1) P concentrations in the surface 10 cm of soil averaged over all months (November through September) for 2008-2009 and 2009-2010.

Treatment	2008-2009	2009-2010
mg kg ⁻¹		
Control	20.5c ^z	14.7c
0.5 – 1.0	28.8a	31.1a
2.0 – 4.0	24.1b	20.2b
DAP	25.4b	18.5b
Analysis of Variance		Prob>F
Month	0.99	<0.01
Treatment	<0.01	<0.01
Month*Treatment	0.04	<0.01

^z Means followed by different letters are significantly different ($P \leq 0.05$).

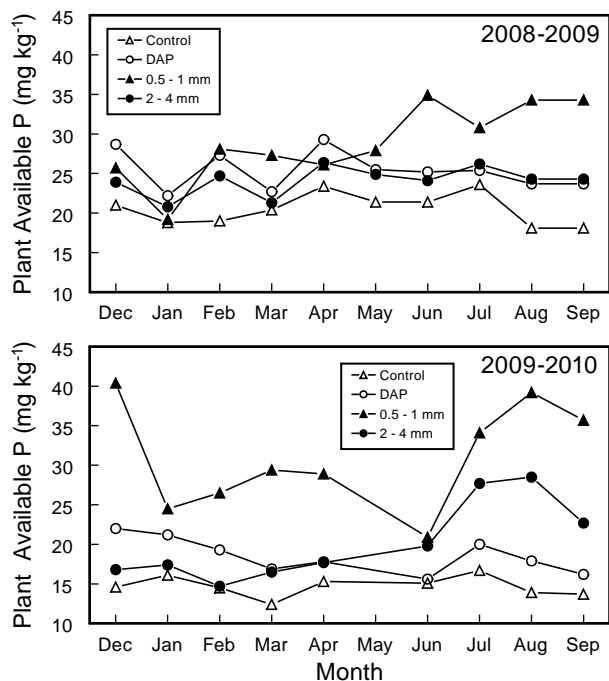


Figure 1. Influence of phosphorus fertilizer source on plant available P (Mehlich 1) in the surface 10 cm of soil from December through September on a Norfolk loamy sand soil in 2008-2009 and a Bonneau sand soil in 2009-2010.

It is not that surprising that the 0.5 – 1.0 mm particle size of recovered P had higher plant available P in the soil than the 2.0 – 4.0 mm particle size of recovered P. This is similar to results found in earlier greenhouse research when particles were mixed with the soil (Bauer et al., 2007). In this field study, we observed that many of the larger-sized particles remained on the soil surface for the duration of the experiments and beyond. This observation prompted us in August of 2010 to collect a portion of the particles that were on the soil surface and analyze them for their P content. We collected particles from each of the plots in the 2008-2009 study (where the fertilizer was applied 21 months earlier) and from each of the plots in the 2009-2010 study (where the fertilizer was applied 9 months earlier). These particles retained most of their P through the months laying on the soil surface. When applied to the plots, the particles had a P content of about 79 g kg⁻¹. Average P content of the particles was 60 g kg⁻¹ (std.dev. 18.4 g kg⁻¹) from the fertilizer applied 21 months earlier and 67 g kg⁻¹ (std.dev. 7.7 g kg⁻¹) from the fertilizer applied 9 months earlier.

On the other hand, it was somewhat surprising that the plant available P in soil from the DAP amended plots was the same as soil from the 2.0 – 4.0

mm size particle amended plots and lower than in soil from plots receiving recovered P in the smaller 0.5 – 1.0 mm particle size (Table 3). Results in Table 2 and inspection of Figure 1 suggest that some of the P in this highly water soluble fertilizer material leached below the 10-cm soil depth that we sampled.

Crop Response. Even though the routine soil test on soil collected across the field conducted two years before establishing the experiment suggested only a medium soil test P level, the localized soil test P levels within the specific experimental plots were higher. Plant available P in the control plots averaged 20.5 mg kg⁻¹ in 2008-2009 and 14.7 mg kg⁻¹ in 2009-2010 (Table 3 and Figure 1). Crozier et al. (2009) articulated the difficulty of assessing new P materials in areas with a history of P fertilizer application. In previous years, the area used for our study was routinely fertilized with P either for experiments or for general crop production.

The history of P applications limited the response of the rye and cotton to the fertilizer treatments. High biomass and P uptake occurred for the rye in 2008-2009. In that year, the application of fertilizer P did not affect P concentration, biomass, or P uptake compared to the control at any sampling date (Table 4). In the second year of the study, the experiment was conducted on a Bonneau sand soil which is more drought-prone and had less plant available P (Table 3) than the Norfolk loamy sand soil used in the first year. On this soil, biomass and P uptake were lower than in 2008-2009. In 2009-2010, application of DAP increased biomass compared to the control at the March and April sampling dates and P uptake at all three sampling dates in that year. In addition to different soil types, planting time could have contributed to the different responses of the two years. Planting of the rye in 2009-2010 occurred a little later in November than in the first year of the study and average daily temperature from planting to the March sampling date was 8.8 °C in 2008-2009 but only 7.3 °C in 2009-2010. Power et al (1980) showed that the deleterious effects of cool soil temperatures on barley (*Hordeum vulgare* L.) were partially alleviated by high levels of available P in soils. The increase in rye growth and P uptake in 2009-2010 that occurred with DAP did not occur for either of the two particle sizes of recovered calcium phosphate. Neither of these treatments influenced growth or P uptake compared to the control in that year.

Table 4. Effect of phosphorus fertilizer source on winter rye P concentration, biomass, and phosphorus uptake at three sampling dates in 2009 and 2010. Means within a column followed by the same letter are not significantly different ($P \leq 0.05$).

Variable	Treatment	2009			2010		
		19 March	9 April	6 May	28 March	14 April	3 May
P Concentration		g kg ⁻¹					
	Control	3.9a	2.5a	1.6a	4.2ab	2.6a	2.1a
	0.5 – 1.0 mm	3.5a	2.8a	1.5a	4.3ab	2.4a	1.7a
	2.0 – 4.0 mm	3.8a	2.8a	1.6a	4.1b	2.6a	2.1a
	DAP	3.8a	2.7a	1.8a	4.9a	2.5a	2.0a
Biomass		kg ha ⁻¹					
	Control	1461a	4275a	7993a	190b	1941b	3426a
	0.5 – 1.0 mm	1641a	4904a	7987a	214b	2474b	4672a
	2.0 – 4.0 mm	1321a	4922a	7626a	219b	1937b	4069a
	DAP	1125a	3705a	5552a	354a	3713a	4568a
P in Biomass		kg ha ⁻¹					
	Control	5.9a	10.6ab	12.9a	0.8b	5.0b	7.1b
	0.5 – 1.0 mm	5.9a	13.7a	12.5a	0.9b	5.9b	7.9ab
	2.0 – 4.0 mm	5.4a	13.9a	12.7a	0.9b	5.1b	8.1ab
	DAP	4.2a	10.0b	10.0a	1.7a	9.1a	8.8a

The higher water solubility of DAP could be the reason for it resulting in higher rye P uptake and biomass than the recovered calcium phosphate at the first two sampling times in the second year of this study. Previous research with conventional tillage found no difference between water-soluble and water-insoluble P fertilizers in their season-long ability to supply P to corn (*Zea mays* L.) when they were broadcast applied and plowed into soil (Webb and Pesek, 1959). Similarly, Owens et al. (1955) found no differences between fertilizers differing in water solubility for

season long plant P uptake. In that study, however; P uptake by plants early in the season (one month after planting) was greater for water-soluble fertilizers than for water -insoluble fertilizers.

Unlike the rye, no differences occurred in either year between the controls and the three fertilizer sources for cotton P concentration, biomass, or total P in the biomass during early flowering (Table 5). Similarly, all three fertilizer application treatments were similar to the controls for seedcotton yield, seed P concentration, and total seed P (Table 6).

Table 5. Effect of phosphorus fertilizer source on cotton P concentration, biomass, and phosphorus uptake during early flowering. Sampling dates were 26 June 2009 and 14 July 2010. Means within a column followed by the same letter are not significantly different ($P \leq 0.05$).

Treatment	2009			2010		
	P Concentration	Biomass	P in Biomass	P Concentration	Biomass	P in Biomass
	g kg ⁻¹	kg ha ⁻¹	kg ha ⁻¹	g kg ⁻¹	kg ha ⁻¹	kg ha ⁻¹
Control	3.2a	1176a	3.7a	3.5a	1086a	3.8a
0.5 – 1 mm	3.6a	979a	3.5a	3.9a	1081a	4.4a
2 – 4 mm	3.4a	1180a	4.0a	3.4a	1040a	3.6a
DAP	3.5a	887a	3.1a	3.7a	1279a	4.7a

Table 6. Effect of phosphorus fertilizer source on P concentration in the cotton seed, seedcotton yield, and total phosphorus in cotton seed in 2009 and 2010. Means within a column followed by the same letter are not significantly different ($P \leq 0.05$).

Treatment	2009			2010		
	P Concentration	Yield	Cotton Seed P	P Concentration	Yield	Cotton Seed P
	g kg ⁻¹	kg ha ⁻¹	kg ha ⁻¹	g kg ⁻¹	kg ha ⁻¹	kg ha ⁻¹
Control	7.3a	4005a	17.7a	6.3a	2470a	9.4a
0.5 – 1 mm	7.1a	3990a	16.9a	6.6a	2370a	9.4a
2 – 4 mm	7.3a	4207a	18.4a	6.9a	2225a	9.2a
DAP	7.2a	4109a	17.6a	6.6a	2563a	9.7a

CONCLUSIONS

This research was conducted to determine the effectiveness of recovered calcium phosphate from manure as a fertilizer source for conservation tillage. The relative ease in processing the material into fertilizer granules suggests that economically competitive P fertilizer products from livestock production facilities may become available to crop producers if advanced manure treatment facilities become more widespread. Because of the higher plant available P in the soil with the small particle size, it appears that if recovered calcium phosphate is used as a P fertilizer source for conservation tillage production it should be applied as a small particle. Long-term studies are needed to assess whether it would provide sufficient P to meet plant requirements when the large particle size is used as a slow-release P source in conservation tillage systems.

DISCLAIMER

Mention of a trade name, proprietary product, or specific equipment does not constitute a guarantee or warranty by the USDA and does not imply approval of a product to the exclusion of others that may be suitable.

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