# Calcium Availability to Runner-type Peanut (*Arachis hypogaea* L.) in the Southeastern United States

by

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#### **Abstract**

Sandy soils in major peanut producing areas in the southeastern United States may be low in plant available Ca. Insufficient Ca supply to developing peanut may result in undeveloped pods and finally low yield, substandard grade, and poor seed quality. Over time peanut varieties change, which may affect Ca absorption physiology, and Ca supplementation products change, which may affect their ability to provide Ca. As a result of these factors, several studies were conducted to reevaluate Ca fertility questions associated with peanut. The objectives of this study were to i) evaluate Ca uptake within peanut maturity classes using the hull-scrape method to determine the potential for early season seed quality assessment; ii) evaluate effect of Ca source and timing on peanut yield, grade, seed Ca, and germination; and iii) evaluate relationships and predictability major soil Ca tests in Coastal Plain soils. Experiments were conducted on a Lucy loamy sand (loamy, kaolinitic, thermic Arenic Kandiudults), a Dothan sandy loam (fine-loamy, kaolinitic, thermic Plinthic Kandiudults), and a Malbis fine sandy loam (fine-loamy, siliceous, subactive, thermic Plinthic Paleudults) in South Alabama. Application of 1120 kg ha<sup>-1</sup> gypsum or lime significantly improved seed Ca concentration and total seed Ca. As peanut size increased, more Ca was absorbed, thus increasing total Ca, but maintaining a relatively consistent seed Ca concentration. Since seed Ca concentration was relatively consistent during 4 wk prior to harvest, seed Ca concentration could be used as an indicator for early season seed quality evaluation. Three commercial types of gypsum had similar effectiveness on supplying Ca to peanut; however, USG 500<sup>®</sup>, a commercially mined gypsum, tended to perform better than

AgriCal<sup>®</sup>, a flue-gas desulfurization (FGD) gypsum, and PCS Wetbulk<sup>®</sup>, a phosphogypsum. When applied at the same rate, lime was as effective as gypsum. Application of Hi-Cal<sup>®</sup>, a liquid Ca fertilizer, could provide adequate Ca to peanut as dry forms of gypsum and lime. There were no significant differences between application of gypsum at planting and early bloom. Split applications of gypsum at early/mid bloom tended to have greater efficacy on improving seed Ca and germination than a single application with an equivalent rate at early bloom. Combined application of lime and gypsum did not show benefits over treatment receiving only lime. Addition of Hi-Cal<sup>®</sup> at mid bloom as a supplement to gypsum applied at early bloom did not significantly improve peanut yield, grade, seed Ca, or germination relative to treatments receiving only gypsum at early bloom. Soil extractable Ca with Mehlich 1 (M1) and Mehlich 3 (M3) was similar and significantly greater than 1N ammonium acetate (AA) and 0.01 M sodium, nitrate (SN). Application of gypsum shortly before soil sampling had minimum effect on correlations among soil Ca by the four tests. However, application of lime led to weaker correlations. Mehlich 1 may overestimate available Ca to peanut in soils recently amended with lime. The AA-extractable Ca showed the best predictability for Ca supplementation for peanut. Mehlich 1-extractable Ca is the second best.

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#### List of abbreviations

AL Alabama K Potassium

ANOVA Analysis of variance kg Kilogram

Ca Calcium m Meter

CEC Cation exchange capacity Mg Magnesium

cm Centimeter mo Month

d Day mm Millimeter

DAP Days after planting USEAP United States Environmental

D. I. H<sub>2</sub>O Deionized water Protection Agency

g Gram wk Week

GA Georgia yr Year

ICP Inductively coupled plasma

#### I. Literature Review

#### Introduction

Peanut (*Arachis hypogaea* L.), belonging to the Leguminosae family, is a self-pollinated, annual herbaceous crop. It is native to South America and is now one of the most popular legume crops grown in subtropical and tropical regions throughout the world. The United States ranks third in peanut production, after China and India (FAS, 2012). In Alabama, which is the third largest peanut producer in the United States behind Georgia and Texas, peanut is a major cash crop with considerable economic importance. On More than 180 million kg of peanut are harvested annually, which generates approximately \$100 million each year for the Alabama economy (Dixon, 2011).

There are four commercial types of peanuts in the United States: Virginia, Runner, Spanish, and Valencia. Runner peanut, which is the primary peanut type grown in Georgia, Alabama, and Florida, accounts for 80% of total U.S. peanut production. Introduction of a runner peanut variety in the early 1960s, "Florunner", resulted in a significant increase in peanut yields (Norden, 1969). Since then, runner peanuts have rapidly gained wide acceptance. Because of their uniform kernel size, runner peanuts can be roasted evenly, making them favorable for producing peanut butter (American Peanut Council, 2012).

Calcium is an indispensable nutrient for peanut seed and pod development. Peanuts lacking Ca may form undeveloped pods called "pops" or have poor germination and vigor. Pods must obtain Ca from the surrounding soil, because Ca is generally immobile in the phloem (Kiesling and Walker, 1982), thus adequate Ca in the pegging zone is essential for proper peanut development. Surface soils of the southeastern U.S. Coastal Plain region tend to be sandy, well-

drained, and friable, providing optimum physical conditions for peanut growth. However, these soils also tend to be naturally low in plant available Ca due to their acidic reaction, low soil organic matter and low cation exchange capacity (CEC). Addition of ground limestone and/or Ca fertilizers, therefore, is a common practice to increase Ca in the pegging zone and improve peanut yield and grade.

Various types of Ca fertilizers, such as ground limestone (CaCO<sub>3</sub>) and gypsum (CaSO<sub>4</sub>), are available to supplement soil Ca. Many experiments were performed to determine the effectiveness of these products in 1970s and 1980s (Hartzog and Adams, 1973; Walker, 1975; Adams and Hartzog, 1979; Hallock and Allison, 1980a; Cox et al., 1982). However, peanut cultivars used in previous studies are no longer cultivated for production. New peanut cultivars may have different development phases that may affect selection and timing of application of a particular Ca fertilizer. Thus, Ca source and timing need to be re-evaluated as Ca fertilizers and peanut varieties change over time.

#### Physiological role of calcium

The significance of Ca in plant growth has been well established. Calcium plays an important role in cell wall and membrane stabilization, cell extension and division, cation-anion balance and osmoregulation, and modulation of certain enzymes. As a second messenger, Ca links environmental and developmental signals to the physiological response of plants (Hawkesford et al., 2012).

Calcium is especially critical in the production of peanut. The consequences of Ca deficiency include an increased number of empty pods, poor quality seeds, seeds with a darkened plumule (Sullivan et al.,1974; Cox et al., 1982; Gascho and Davis, 1994), and high incidences of

pod rot (Garren, 1964; Walker and Csinos, 1980), resulting in severe loss in yield and substandard grade. Peanut seeds produced in Ca-deficient conditions have poor germination and vigor due to the degradation of the primary root before plumule emergence (Burkhart and Collins, 1941). Calcium also affects the water status and membrane permeability of peanut leaves. Under moisture stress conditions, the extent of membrane damage and loss of water is less severe in leaves of plants grown with higher levels of Ca compared to those not receiving a Ca supplement (Chari et al., 1986).

#### Calcium requirement during peanut development stages

The growth of peanut consists of two stages: vegetative and reproductive. Vegetative growth, including stem elongation and leaf development, is generally completed 110 days after planting (DAP) provided the moisture condition is optimal. Reproductive growth involves flowering, fertilization, pegging, and pod development. Flowering normally begins 25 to 35 DAP, and maximum flowering occurs 4 to 6 wk following the appearance of the first flower (Weeks et al., 2000). Peanut seed development is distinctive from other plants. After pollination and fertilization, the ovary develops into a stalk-like structure called a "peg", and penetrates the soil (Smith, 1950). The entrance of the peg into the soil occurs about 14 d after appearance of flowers (Pickett, 1949). The peg then enlarges to form an underground pod and finally a mature peanut in the pegging or fruiting zone.

After the peg enters the soil, the developing pod has no functional transpiration, thus it has no direct access to root-absorbed and xylem-transported nutrients (Bledsoe et al., 1949; Wiersum, 1951; Skelton and Shear, 1971; Kvien et al., 1988). Lack of xylem-transported nutrients combined with the low physiological mobility of Ca in the phloem forces pods to

acquire Ca from the surrounding soil (Brady, 1947; Kiesling and Walker, 1982). Calcium enters the seed by diffusion directly from soil into the hull. It is then transported to the seed via the funiculus (Sumner et al., 1988; Smal et al., 1989). It is thus important to maintain a high concentration of Ca in the pegging zone in order to create a gradient of Ca towards the pod (Smith et al., 1993).

The requirements of Ca in the pegging zone for optimum pod yield are considerably higher than those required for proper vegetative growth (Comber, 1959; Wolt and Adams, 1979); therefore, it is necessary to determine the Ca uptake period of the peanut. Brady (1947) found that the critical period for Ca absorption was 15 to 35 d after pegging, whereas Gascho and Davis (1995) found the critical periods for Ca absorption were about 20 to 80 d following entrance of the peg into the soil. Mizuno (1959) determined that 92% of Ca was absorbed by the pod during the first 20 d after peg has entered the soil. However, recent research using currently produced peanut cultivars suggests that Ca is absorbed by pods throughout development until final maturity (Howe et al., 2012).

#### Calcium supplements

Addition of Ca to soils that are low in Ca can improve peanut yield and grade (Adams et al., 1993), as well as germination and vigor (Sorenson and Butts, 2008). Lime, gypsum, and liquid Ca fertilizers are the most commonly used Ca supplements. They have various solubilities, plant availabilities, and longevities such that provides their own advantages and disadvantages.

Lime can increase soil pH, alleviate Al and Mn toxicities, and increase pegging zone Ca (Hartzog and Adams, 1973). Adams et al. (1979) found that application of lime could improve yield and the percentage of sound mature kernels (SMK). Lime has a long residence time in the

soil compared to other Ca sources. When lime was disked into the top 15 cm of soil, it supplied Ca to the pegging zone for up to 20 months (Reed and Brady, 1948). However, the low solubility of lime limits Ca availability for pod formation, especially under dry conditions.

Gypsum is the most common amendment for Ca when soil pH is adequate for peanut production, but soil Ca is limiting. It has been shown to improve yield, grade, and germination (Hallock and Allison, 1980a), but applications of gypsum at high rates may reduce availability of K and Mg in the pegging zone (Alva and Gascho, 1990; Hallock and Allison, 1980b). Gypsum has a much higher solubility in water (2.6 g L<sup>-1</sup>) than lime (can you give a value??). This could result in shorter residence time for gypsum in the soil due to leaching following rainfall and irrigation. Gypsum was also found to improve soil physical and chemical properties. Soils with added gypsum have been reported to have less surface crusting and compaction, greater water infiltration and water holding capacity, greater aggregate stability, and less water runoff and erosion compared to soils receiving no gypsum (Clark et al., 2001).

Liquid Ca fertilizer, which can be applied with or without irrigation, is an alternative to lime and gypsum that is frequently marketed to peanut producers. However, many studies have reported lower effectiveness of liquid Ca fertilizers compared to dry forms of gypsum and lime (Hartzog and Adams, 1973; Hallock, 1975; Walker, 1975; Adams and Hartzog, 1979). Differences between dry and liquid Ca fertilizers are probably due to the lower total Ca input with application of liquid Ca compared to gypsum (Walker, 1975) or lime. Another reason for reduced efficacy of liquid fertilizers is the greater leaching potential associated with highly soluble liquid forms compared to solid forms, especially on sandy soils with low CEC.

#### Currently available Ca fertilizers

A variety of liming materials, such as calcitic lime, dolomitic lime, and hydrated lime, are available to peanut producers. These materials differ in purity, particle size, and calcium content, influencing the method and timing of the liming application. Acid, sandy soils in the Southeast may be low in Mg, so dolomitic lime is widely used to increase soil pH as well as provide Ca and Mg.

Many types of gypsum are available for peanut production including geological and anthropogenic sources. Naturally mined gypsum, also known as "landplaster", has long been used as a source of Ca for peanut production. The United States is the second largest landplaster producer after China (USGS, 2014). USG 500 landplaster™, manufactured by the U.S. Gypsum Corporation (Chicago, IL), is popular among peanut producers in the Southeast. According to product information, USG 500 landplaster™ is a natural, high-purity crystalline gypsum material that contains 21% Ca (dry weight, dw).

Synthetic gypsum products, such as flue gas desulfurized (FGD) gypsum and phosphogypsum, have drawn attention in recent years as alternatives to geologic gypsum. The FGD gypsum, which is nearly chemically identical to naturally mined gypsum, is generated as a byproduct of coal-fired electric power plants. AgriCal<sup>®</sup>, containing approximately 26% Ca (dw), is a type of FGD gypsum manufactured by the Southern Company (Albany, GA). Grichar et al. (2002) showed that FGD gypsum did not significantly improve yield or grade of runner peanut over untreated control treatment in Texas. However, few studies have determined the effect of FGD gypsum on runner peanut production in the Southeast. More research will be needed to evaluate effectiveness of FGD gypsum on various soil types and peanut varieties.

Phosphogypsum, which is a byproduct of processing phosphate rocks into fertilizer with

sulfuric acid, is a gypsum with a low concentration of phosphorus. It has a pH that is normally between 4.5 and 5.0 (Korcak, 1998). The composition of phosphogypsum varies depending on the source of phosphate ore and the process for manufacturing phosphoric acid (Mays and Mortvedt, 1986). The major problem with using phosphogypsum in agriculture is finding sources that do not contain radioactive radium and radon. The U.S. Environmental Protection Agency (USEPA) has permitted controlled use of phosphogypsum in agriculture if radium-226 levels are <10 pCi g<sup>-1</sup>. This restriction on the maximum radium radioactivity prohibits the use of some phosphogypsum mines, yet does not impact phosphogypsum from northern Florida or North Carolina, which generally have lower levels of radium-226 (Korcak, 1998). PCS Wetbulk<sup>®</sup>, which contains approximately 20% Ca (dw) according to its product label, is the type of phosphogypsum product manufactured in White Springs, FL. Many studies have shown that phosphogypsum can alleviate some detrimental effects of subsoil acidity on plant growth (Alva and Sumner, 1989; Alva and Sumner, 1990; Alva et al., 1990; Caldwell et al. 1990). However, few studies have been conducted to test the efficacy of phosphogypsum on peanut production against other types of gypsum.

In addition to FGD gypsum and phosphogypsum, synthetic gypsum also includes materials such as citrogypsum (a byproduct of citric acid production), fluorogypsum (a byproduct from the production of hydrofluoric acid from fluorspar), and Titanogypsum (a byproduct from the production of titanium dioxide). Benefits and environmental risks of these materials are still unknown and need to be carefully evaluated before agricultural utilization.

Some liquid Ca fertilizer products are also available in the market. These products give the peanut growers a liquid Ca choice that is soil applied as a replacement for gypsum or lime. Hi-Cal<sup>®</sup> liquid Ca, which contains 12-13% Ca by weight (w/w), is a clear calcium chloride (CaCl<sub>2</sub>)

solution with neutral pH (TETRA Technologies, Inc., The Woodlands, TX). TigerCal 30<sup>TM</sup> is a concentrated Ca product. It contains 30% (w/w) CaCO<sub>3</sub> that is chelated with 1% humate (Tiger Industries, Inc., Bristol, RI). CaTs<sup>®</sup> is a neutral to basic, chlorine-free, clear solution, containing 6% Ca and 10% S (w/w) derived from calcium thiosulfate (CaS<sub>2</sub>O<sub>3</sub>). It can be foliar applied or soil applied. However, CaTs<sup>®</sup> is not compatible with phosphate fertilizers according to the product information (Tessenderlo Kerley, Inc., Phoenix, AZ). These relatively new liquid Ca products are claimed "readily available" to plants by manufacturers; however, in many cases efficacy of them for peanut is still unknown in the southeastern United States.

#### Effect of particle size and solubility on gypsum availability

The solubility of gypsum increases with increasing particle surface area (smaller partice size). Studies have shown that fine gypsum has a higher rate of dissolution than coarse gypsum in deionized water or diluted salt solutions (Alva et al., 1989; Bolan et al., 1991; Keisling and Walker, 1978). Authors assumed that the fine gypsum material was more effective in the early season than the coarse material, but the coarse material may persist longer and supply Ca to the pegging zone over a longer period than the fine gypsum material. However, field experiments that compared gypsum materials having different particle sizes demonstrated that fine and coarse gypsum were equally effective in supplying Ca to the pegging zone (Daughtry and Cox, 1973; Keisling and Walker, 1978). Keisling and Walker (1978) reported that the relative effectiveness of different gypsum materials was significantly impacted by the rainfall pattern. Infrequent but high magnitude rainfall tends to reduce soil Ca concentration near the soil surface, whereas several small rainfall events could result in accumulation of Ca near the soil surface (Keisling and Walker, 1978).

Only a few studies have evaluated the effects of various physical forms of gypsum (e.g., granular, powder, crystalline, etc.) on peanut yield. Of these, none have found a significant difference in peanut yield due to the particle size of the gypsum material applied (Daughtry and Cox, 1974; Reed and Brady, 1974; Hallock and Allison, 1980a; Alva et al., 1989). A more detailed investigation of the relationship between solubility and Ca availability over the peanut development phase may be needed to fully understand it.

#### Effect of calcium source and timing on peanut yield

The management goal for Ca is to ensure Ca availability throughout the growing season, especially when peanut is grown for seed. Since availability of Ca relies on the type of supplement, it is necessary to optimize the timing of application of Ca sources based on their properties as well as the targeted availability to the developing peanut.

A limited number of studies have been conducted to determine effect of Ca source on peanut yield. Daughtry and Cox (1974) compared the relative effectiveness of regular landplaster, granular landplaster, and phosphogypsum, and concluded that there was no significant difference among the three materials on increasing soil Ca and peanut yield. However, Ca content in the top 20 cm of soil was 960 kg ha<sup>-1</sup> in this study, which should not be a limiting factor for peanut production (Colwell and Brady, 1945; Hartzog and Adams, 1973). Grichar et al. (2004) found that FGD gypsum and a locally available "agricultural gypsum" were equally effective on increasing runner-type peanut yield and grade on soils with intermediate (96 to 140 mg kg<sup>-1</sup>) Mehlich 1-extractable Ca in Texas.

Studies comparing lime sources (e.g., calcitic and/or dolomitic) with gypsum have had inconsistent results. Sullivan et al. (1974) found that application of lime before planting had no

effect on peanut yield, but application of gypsum at early flowering could significantly improve peanut yield and grade. In contrast, Gascho et al. (1993) reported that calcitic and dolomitic limestone applied before planting were equally effective for the purpose of increasing peanut yield, whereas gypsum applied at full bloom did not significantly increase peanut yield. Adams and Hartzog (1980) found differences in yield between dolomitic and calcitic lime. However, low subsoil Mg, rather than Ca, was suggested to be responsible for the differences among the sources, and lime and gypsum were found to be equal sources of Ca (Adams and Hartzog, 1980).

Effectiveness of liquid Ca fertilizers has also been analyzed. Adams and Hartzog (1979) compared a 50% lime slurry with two types of dolomitic limestone, indicating application of dry lime resulted in higher peanut yield than the lime slurry. Hartzog and Adams (1973) examined the efficacy of MagiCal®, a liquid gypsum product manufactured by Standard Spray and Chemical Company (Lakeland, FL). This type of foliar-sprayed Ca did not increase peanut yield or grade on low Ca soils when gypsum did improve yield and grade. Walker (1975) compared two types of liquid Ca fertilizers, MagiCal® and a chelated Ca solution with gypsum and found that gypsum improved yield and grade greater than either of the liquid fertilizers. Hallock (1975) compared the effectiveness of five liquid Ca fertilizers: THIS® solution, derived from CaCl<sub>2</sub> (Stoller Chemical Company, Houston, TX); MagiCal® solution, derived from CaSO4; CaB® solution, derived from CaNO<sub>3</sub> with 0.5 % boric acid (Leffingwell Chemical Company, Brea, CA); CaS® solution, derived from CaSO<sub>4</sub> with additional sulfur (Echol Chemical Formulators Inc., Lakeland, FL); and CaNO<sub>3</sub> solution (Wilson & Geo. Meyer & Company, San Francisco, CA) on yield and grade of Virginia type peanuts on four different soil types during 1971 through 1974. Application of these liquid Ca fertilizers at early flowering stage at recommended rates by manufacturer did not improve yield or grade compared to the control treatment. Only when both

gypsum and liquid Ca fertilizer were applied did yield and grade significantly increase compared to the control treatment, but there was no significant increase in gross crop value over the treatment receiving only gypsum.

There are few studies evaluating the timing of Ca applications, and results are inconsistent. Reed and Brady (1948) found application of gypsum at early bloom provided more available Ca than application at seedling emergence, while Hallock and Allison (1980a) showed no difference in yield among three gypsum application times, at planting, 30 DAP, and early flowering, although there was a trend of higher yield with application at early bloom. Hallock (1975) found that when applied at early flowering, the two tested liquid Ca fertilizers, THIS® and CaNO3 solution, produced significantly higher peanut yield, grade, and gross crop value than applied at full-flower stage. In addition, multiple applications of MagiCal® or CaNO3 solutions generally were superior to a single application equivalent to the total of the multiple applications (Hallock, 1975).

#### Methods used to extract Ca, K, and Mg from the soils

Soil testing is an important part of nutrient management. The primary goal of soil testing is to determine relationships between soil chemical properties and crop growth. Since Ca plays a key role in peanut production, several methods have been employed to extract Ca from soil to represent available Ca for developing pod.

The most commonly used Ca extractant in the Southeast is the Mehlich 1 extractant (M1), which is a mixture of 0.05 M HCl and 0.025 M H<sub>2</sub>SO<sub>4</sub> (Mehlich, 1953). It is a versatile soil extractant that is used to determine the amounts of P, Ca, K, Mg, and several other soil nutrients. It works well for acidic, low CEC soils, which includes almost all soils in the Southeast. The

Mehlich 3 extractant (M3), composed of 0.2 N acetic acid, 0.25 N ammonium nitrate, 0.015 N ammonium fluoride, 0.013 N nitric acid, and 0.001M EDTA, was developed to cover a wide range of soils including neutral to alkaline soils (Mehlich, 1984). This extractant can simultaneously extract soil P, Ca, Mg, K, as well as some micronutrients, and thus significantly improve efficiency of soil testing. Another extractant that has been widely used for extraction of exchangeable cations is 1 N neutral ammonium acetate (AA). Seventeen states, primarily in the north central and western United States, have adopted this extractant for exchangeable cations (Allen and Johnson, 1994). Many studies have shown strong correlations among these three extractants for individual nutrients over a wide range of soils, and conversion equations have been developed to convert the results obtained with a certain extractant to the other one (Hartzog et al., 1973; Mehlich, 1984; Michaelson et al., 1987; Sims et al., 1989; Beegle and Oravec, 1990; Evans and McGuire, 1990; Gascho et al., 1990; Gartley et al., 2002; Mylavarapu et al., 2002; Wang et al., 2004; Franklin and Simmons, 2005).

In order to improve the predictability of Ca requirements by peanut, Smal et al. (1988) developed a new extractant, 0.01 M sodium nitrate solution (SN). Their results suggest that this extractant closely measured the readily available Ca in the soil solution while M1 extracted some unavailable Ca. Smal et al. (1988) also reported that peanut yield, grade, and pod rot were better related to Ca extracted with SN than M1. However, Alva et al. (1989) found peanut yield was better related to M1 rather than SN. Since evaluation of the relationships between SN and other extractants in Coastal Plain soils is limited for peanut, further study is necessary for determination of correlations among extractants and selection of methods used for predicting the supplemental Ca requirement for peanut.

Recommendations for Ca supplementation for peanut production in the Southeast are currently based on soil test Ca by M1. However, evidence shows that M1 extracts more soil Ca following lime treatment than following gypsum treatment (Alva et al., 1990; Csinos et al., 1982; Csinos et al., 1983), indicating M1 is an adequate index only when no lime was applied or lime was applied well in advance of soil sampling. Alva et al. (1989) also reported that soils with higher AA extractable Ca did not guarantee higher peanut yield compared to soils with low AA extractable Ca, suggesting that AA did not necessarily represent available Ca to developing peanut. Alva et al. (1990) compared relationships between M1- and SN- extractable Ca on Coastal Plain soils. Results indicated that relationships between SN and M1 extractable Ca were better for gypsum treatment ( $r^2 > 0.6$ ) than for lime treatment ( $r^2 < 0.2$ ). More research is needed to fully understand effects of Ca source on extracted Ca.

#### **Summary**

Calcium is an essential nutrient for proper peanut development. Surface soils in the Southeast are optimum for peanut production but naturally low in Ca. Evaluating seed quality or Ca fertilization needs during early season may be helpful to peanut producers. Recent research showed that maturity class of the peanut, based on the hull scrape method, had no effect on seed Ca concentration, so seed Ca concentrations in early season peanuts may reflect final concentrations in peanuts, indicating it can be used as an index for early season seed quality assessment. However, these results need to be verified and evaluated over a range of Ca growth conditions.

Addition of Ca supplements, such as lime, gypsum, and liquid Ca fertilizers, is a normal practice to improve peanut yield and grade. Each source of Ca has advantages and disadvantages

to peanut producers. Lime stays in the pegging zone longer but creates a smaller Ca gradient to the peanut. Gypsum creates a larger Ca gradient, but it can leach out of the pegging zone after significant rainfall. Liquid Ca fertilizers supply very little total Ca when compared to lime or gypsum, but they are readily available to the developing pod. As Ca products and peanut cultivars change over time, selection and timing of application of a particular Ca fertilizer for maximum peanut yield, grade, and seed quality need to be re-evaluated.

Accurate evaluation of soil Ca is critical to determine the need for Ca fertilization. Many different methods have been employed to extract Ca from the soil, including M1, M3, AA, and SN. Many studies have compared the M1, M3, and AA extractants and found them to be significantly correlated ( $r^2 > 0.9$ ) for the entire suite of plant nutrients. However, research also found the amount of Ca extracted showed greater variation than did other nutrients. Some research supports that this may be due to previous application of Ca fertilizers that remain in the soil and react differently from native soil Ca. Other researchers have found that extraction of Ca with SN improved predictability of Ca requirement for peanuts compared to the M1 extractant. Correlation of the SN method to other soil testing methods and determining effect of prior Ca amendments on soil test values could improve Ca fertilization recommendations.

In order to address these needs by peanut producers, studies were conducted to 1) evaluate Ca uptake within the peanut maturity classes using the hull scrape method to determine the potential for early season seed quality assessment; 2) evaluate effect of Ca source and timing on peanut yield, grade, seed Ca, and germination; and 3) compare different methods used to extract soil Ca and evaluate the influence of Ca amendments on extracted Ca, K, and Mg.

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# II. Calcium Uptake of Peanut Seeds by Developmental Stages

## **Abstract**

Calcium is an indispensable nutrient for proper peanut pod development and indicative of seed quality; therefore, it is important to know Ca uptake by developmental stages for optimum timing of Ca application. This study was conducted to evaluate the effects of sampling time, maturity class, and Ca treatment on seed weight, seed Ca concentration, and total seed Ca in order to develop a pre-harvest seed quality assessment. A field trial was conducted at Wiregrass Research and Extension Center in Headland, AL, in 2012 under non-irrigated conditions using two of the most popular cultivars grown in the Southeast, Georgia-06G and Georgia Greener. Treatments included application of flue gas desulfurized (FGD) gypsum at rates of 560 and 1120 kg ha<sup>-1</sup> at early bloom, lime at 1120 kg ha<sup>-1</sup> at planting, and an untreated control. Peanut maturity classes were determined using the hull scrape method. Results showed that addition of gypsum and lime significantly increased seed weight. Application of 1120 kg ha<sup>-1</sup> gypsum at early bloom or lime at planting significantly improved seed Ca concentration and total seed Ca compared to the untreated control, whereas 560 kg ha<sup>-1</sup> gypsum at early bloom did not. When peanut seeds mature from yellow to black stage, more Ca was absorbed as seed size increased, thus total Ca in seed increased, but a relatively consistent seed Ca concentration was maintained at each sampling time. Therefore, seed Ca concentration has the potential to be used for evaluation of seed quality in early season, allowing for possible management to improve total seed Ca prior to harvest.

## Introduction

Proper development of peanut requires a sufficient Ca supply in the pegging zone. Addition of Ca supplements (e.g., lime and gypsum) to low Ca soils in the southeastern United States has been found to increase peanut yield, grade, and germination (Adams et al., 1993; Smal et al., 1989; Sorenson and Butts, 2008). Seed Ca concentration and germination rate are highly correlated on runner peanut (Adams and Hartzog, 1991; Adams et al., 1993). Peanuts lacking Ca often have black-ended plumule and consequently low germination rate. It would be beneficial if seed Ca concentration could serve as an indicator of germination or seed quality.

Optimum pod yield requires significantly higher amounts of Ca in the pegging zone than proper vegetative growth (Comber, 1959; Wolt and Adams, 1979), thus timing of different Ca sources could benefit from determination Ca uptake within different pod development stages. The hull scrape method, developed by Williams and Drexler (1981), is commonly used to determine peanut maturity class distribution and to assess when the majority of peanuts are mature (brown or black) and ready for harvest. This method reveals the colored mesocarp that develops according to the following progression as peanuts mature: white, yellow 1, yellow 2, orange, brown, and black.

The maturity class, based on hull scape method, has been correlated with changes in seed physical and chemical properties. Kim and Hung (1991) reported that seed moisture content decreased with maturity as the mesocarp color changed from yellow 1 to black. They also found that seed size and oil content increase from yellow 2 to orange class, but remain approximately the same as peanuts progress into brown and black classes. However, there are few experiments that have determined the maturity class for Ca uptake by peanut. Early studies indicate most of the Ca was absorbed within 30 d after pegging (Brady, 1947; Mizuno, 1959). Recent work by

Howe et al. (2012) found preliminary evidence that the seed absorbed Ca as it enlarged until final maturity, but the Ca concentration in seed is relatively consistent; therefore, seed Ca concentrations in early season peanuts may reflect final concentrations provided that soil Ca concentrations do not change dramatically. However, these results were based on a single soil Ca condition and may not reflect distribution under various Ca growth conditions. Further study is needed to verify these results and to determine the earliest possible sampling period to precisely estimate seed Ca concentrations. The objective of the current study was to evaluate the effects of cultivar, sampling time, maturity class, and Ca treatment on seed weight, seed Ca concentration, and total Ca in seed over a range of Ca conditions in order to develop a preharvest seed quality assessment.

#### **Materials and Methods**

A field experiment was conducted at Wiregrass Research and Extension Center (WREC) in Headland, AL (31.36 °N, 85.32 °W), in 2012, under non-irrigated conditions. Soil type in this study is Lucy loamy sand (loamy, kaolinitic, thermic Arenic Kandiudults). The field experiment was arranged as a split-plot design with 4 replications. The main plot factor was the Ca treatment while the sub-plot factor was peanut cultivar. Application of flue gas desulfurized (FGD) gypsum at rates of 560 and 1120 kg ha<sup>-1</sup> at early bloom, lime at 1120 kg ha<sup>-1</sup> at planting, and an untreated control treatment were compared. Two of the most popular cultivars grown in the Southeast, 'Georgia-06G' and 'Georgia Greener', were evaluated. No fertilization other than the Ca treatments were applied. Typical management practices for peanut production (e.g., herbicide and fungicide) were followed. Rainfall data in Headland, AL, was collected by

Alabama Mesonet at the Headland weather station (Agricultural Weather Information Service, Inc., Auburn, AL) located within 2 km from WREC.

Pegging zone soil samples were collected from each sub-plot before planting using a handheld soil probe. Approximately 14 cores were taken per sub-plot, combined in a bucket, and mixed well to provide a composite sample. Soils were dried at 60 °C for 48 h and finely ground using a mortar and a pestle to pass a 2-mm sieve. Soil pH was determined according to method by Hue and Evans (1986). Briefly, 10 g soil was mixed with 20 mL deionized water (D.I. water) and shaken for 10 min. After settling 30 min, pH was measured using a pH meter (Sevencompact<sup>TM</sup> pH/Ion meter S220, Mettler-Toledo LLC, Columbus, OH). Soil Ca, Mg, and K concentrations were determined using the Mehlich 1 extraction (Mehlich, 1953). Briefly, 5 g soil was shaken with 20 mL Mehlich 1 extractant solution for 5 min, centrifuged at 1000 rcf (IEC Model K centrifuge, International Equipment Company, Needham Heights, MA) for 10 min, and then filtered using Whatman No. 40 filter paper (GE Healthcare Life Sciences, Pittsburgh, PA). The filtrate was analyzed with inductively coupled plasma (ICP) spectroscopy (Spectro Ciros CCD, side on plasma, SPECTRO Analytical Instruments Inc., Kleve, Germany).

Peanut samples were taken 4, 2, and 0 wk before harvest from each sub-plot. Sixteen peanut plants were hand pulled from each sub-plot. Pods were removed from the plants using a scrubber, pressure washed to reveal colors of mesocarp, and sorted by maturity classes according to the hull scrape method by Williams and Drexler (1981). Peanuts classified as "white" were discarded, because peanuts within this class were too small to be considered as "harvestable peanut". The peanuts were then shelled, dried at 60 °C for 48 h, counted, weighed, and finely ground in a coffee grinder (Hamilton Beach Inc., Picton, Canada). Seed Ca was determined by microwave digestion using a Mars Xpress microwave (CEM Corp., Matthews, NC) using the

modified EPA 3051A procedure (USEPA, 2007) followed by Ca determination by ICP. In detail, 1 g finely ground peanut sample was pre-digested overnight in 10 mL 30% (v/v) hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) and then microwave digested for 15 min in 10 mL concentrated nitric acid (HNO<sub>3</sub>). The final digested material was filtered using Whatman No. 40 filter paper, and the volume was adjusted to 100 mL with D.I. water before analysis by ICP.

Mixed models methodology as implemented in SAS PROC MIXED were used to analyze the data (Littell et al., 2006). Cultivar, Ca treatment, and maturity class were treated as fixed effects, and block was the only random effect. Sampling time was treated as repeated measurement. To test for differences among main effects and their interactions, the Kenward-Roger (KR) adjustment was applied to the least square means (LSMEANS) statement of SAS software.

# **Results and Discussion**

Initial soil pH for this study was 5.6, while the initial soil Ca, K, and Mg in the pegging zone was 164, 23, and 24 mg kg<sup>-1</sup>. Rainfall received in this study during the peanut growing season (May to October) in 2012 was 566 mm, which is considered normal rainfall compared to the 10 yr mean rainfall of 576 mm in Headland, AL.

Seed weight, seed Ca concentration, and total seed Ca were evaluated for main effects and interactions due to cultivar, treatment, maturity class, and sampling time. Cultivar was a significant main effect for seed weight and seed Ca concentration, but not for total seed Ca (Table 1). The seed weight of Georgia-06G was significantly greater than Georgia Greener, but the seed Ca concentration was significantly lower in Georgia-06G (Figure 1). Kvien et al. (1988) showed that relatively small pod size favors high Ca concentration in seeds. The current

study reached the same conclusion. However, work published by Tillman et al. (2010) argued that Ca concentration of runner-type peanut seeds was mainly affected by genotype, not seed size. Further studies using peanut cultivars that have similar seed size are necessary to better understand this issue.

Many studies have reported that Ca requirements of the large-seeded Virginia-type cultivars is greater than the relatively small-seeded runner-type varieties (Middleton et al., 1945; Walker et al., 1976; Walker and Keisling, 1978; Cox et al., 1982; Gaines et al., 1989). The two runner-type cultivars used in this study, large-seeded Georgia-06G and medium-seeded Georgia Greener, had similar total Ca in seed, indicating similar Ca absorption capability by the two varieties (Figure 1). Lower seed Ca concentration in Georgia-06G can be attributed to the diffusion effect caused by its larger seed size. There was no interaction among cultivar and other factors including treatment, maturity class, and sampling time; thus data for Georgia-06G and Georgia Greener were combined for all other evaluations.

Maturity class, sampling time, and Ca treatment notably affected (P < 0.001) seed weight, Ca concentration, and total Ca in seed. No interactions among Ca treatment and other factors were observed on seed weight (Table 1). Application of Ca amendments notably increased seed weight compared to the control treatment, but no significant differences were observed among the three Ca treatments (Figure 2).

A sampling time × Ca treatment interaction was noted on seed Ca concentration and total Ca in seed, but not on seed weight (Table 1). Within each sampling period, application of lime or gypsum at 1120 kg ha<sup>-1</sup> notably improved seed Ca concentration and total Ca in seed above the control and gypsum at 560 kg ha<sup>-1</sup> treatments (Table 2). There was no significant difference in seed Ca concentration and total seed Ca between lime and gypsum when applied at 1120 kg ha<sup>-1</sup>.

This finding is supported by Adams and Hartzog (1980) that demonstrated lime and gypsum were equal source of Ca. However, at certain sampling times (e.g., 2 wk before harvest), seed Ca concentration and total Ca in seed differed between lime and gypsum at a rate of 1120 kg ha<sup>-1</sup>. When this occurred, generally the lime treatment resulted in greater seed Ca concentration or total Ca in seed. Application of 560 kg ha<sup>-1</sup> gypsum did not significantly improve seed Ca concentration or total Ca in seed compared to the control (Table 2). Overall result of a study performed at 14 site-years suggested that application of 560 kg ha<sup>-1</sup> gypsum significantly increased seed Ca concentration (Howe et al., 2012). Inconsistent results between this study and work by Howe et al. (2012) is likely due to different moisture conditions during the study. Water stress affects seed and hull Ca concentrations through its effect on Ca diffusion into the pods (Sumner, 1988; Kvien et al., 1988). The current study was performed under non-irrigated conditions; however, half of the 14 tests conducted by Howe et al. (2012) were irrigated and some of the tests under non-irrigated conditions occurred in relatively wet years. These factors likely helped overcome diffusion limitations and increased Ca availability to developing pods.

Both seed Ca concentration and total seed Ca increased from 4 to 2 wk before harvest. Seed Ca concentration and total seed Ca were similar at 2 and 0 wk before harvest (Table 2). Total seed Ca and seed Ca concentration significantly declined from 2 to 0 wk before harvest in the lime at 1120 kg ha<sup>-1</sup> at planting treatment, while other treatments remained approximately the same (Table 2). These results indicate that Ca absorption may be limited during this time; however, this is likely due to differences in moisture during these periods. Poor Ca availability in the lime treatment during this period may be attributable to dry conditions that limit absorption. During the 2 wk prior to harvest, only 0.46 mm rainfall was received. Gypsum solubility (2.6 g L<sup>-1</sup>) is greater than lime (0.01 g L<sup>-1</sup>), which reduces limitations to diffusion;

therefore, seed Ca concentration and total seed Ca increased during the 2 wk before harvest in the gypsum at early bloom at 1120 kg ha<sup>-1</sup> treatment (Table 2).

The treatment × maturity class interaction showed significant effect on seed Ca concentration and total seed Ca, but not on seed weight (Table 1). Seed Ca concentration and total seed Ca of all the maturity classes were notably improved with application of lime or gypsum at 1120 kg ha<sup>-1</sup> compared to the control and gypsum at 560 kg ha<sup>-1</sup> treatments.

Application of 560 kg ha<sup>-1</sup> gypsum significantly improved seed Ca concentration in the orange class, but for all the other maturity classes, seed Ca concentration and total seed Ca for gypsum at 560 kg ha<sup>-1</sup> were similar to the control (Table 3). When applied at 1120 kg ha<sup>-1</sup>, lime and gypsum were equally effective on increasing seed Ca concentration and total seed Ca of all maturity classes, except the brown class. Lime produced higher seed Ca concentration and total seed Ca than gypsum for the brown class (Table 3). From yellow 1 to black class, seed Ca concentration only slightly increased, while total seed Ca almost doubled. This trend applies to all the Ca treatments in this study (Table 3).

There was a significant maturity class × sampling time interaction on seed weight, seed Ca concentration, and total seed Ca (Table 1). Seed weight significantly increased within the 4 wk period before harvest. Seed weight at 4, 2, and 0 wk before harvest were 453, 564, and 632 mg seed<sup>-1</sup>, respectively. At each sampling time, seed weight increased from yellow 1 to brown class (Figure 3). Mature seeds (brown and black classes) had higher seed Ca concentration than immature seeds (yellow 1 and yellow 2 classes); however, seed Ca concentration did not differ greatly throughout the developmental stages compared to seed weight and total seed Ca (Figure 3). As seed size increased during the developmental period, more Ca was absorbed, thus increasing total seed Ca accordingly but not greatly changing the Ca concentration (Figure 3).

Very few studies have determined Ca uptake by periods. Brady (1947) and Mizuno (1959) found that the critical period for Ca uptake occurred within 15-35 d after pegging. During this period, they found that up to 92% of Ca was absorbed by pods. Gascho and Davis (1995) found that the critical periods for Ca absorption start about 20 d after pegging and may extend another 60 d. However, recent study by Howe et al. (2012) showed that peanut pods absorb Ca throughout the whole season until final maturity. This finding was substantiated by the current study under different Ca growth conditions. Because seed Ca concentration was relatively consistent during the 4 wk period leading to harvest, it has the potential to be used as an indicator of early season seed quality assessment.

## **Conclusion**

The large-seeded Georgia-06G and medium-seeded Georgia Greener were found to differ in their seed Ca concentration; however, their ability to accumulate Ca in seed was similar across a range of Ca growth conditions. The genetic variability in Ca accumulation may be used by peanut breeders to produce cultivars which require less Ca supplementation. In this study, application of 560 kg ha<sup>-1</sup> gypsum did not supply adequate Ca to developing pods whereas gypsum or lime applied at 1120 kg ha<sup>-1</sup> did. However, the relatively high rainfall received during the study is likely to improve Ca availability in the pegging zone by lime. Production of peanuts without irrigation, especially in drier years, can benefit most from gypsum applications since it has a higher solubility and thus lower limitation of diffusion than lime. Therefore, gypsum applied at 1120 kg ha<sup>-1</sup> at early bloom is recommended for peanut growing under non-irrigated conditions.

During the 4 wk prior to harvest, total seed Ca increased across developmental stages at each sampling time, indicating Ca was absorbed by seed throughout development until harvest. Therefore, it is important to maintain an adequate Ca supply in the pegging zone during pegging until harvest. This may also provide useful information regarding timing of Ca applications. A single application of gypsum at early bloom is a common practice among peanut producers. However, applied gypsum may be readily leached out of the pegging zone after heavy rainfall, making it unable to meet the Ca requirements by developing pods in the late season. Additional Ca supplementation in mid to late season or split applications of gypsum may be beneficial compared to a single application in the early season. Unlike total seed Ca, seed Ca concentration was relatively consistent across all maturity classes at each sampling time within the 4 wk period leading to harvest; therefore, it may be used as an indicator of pre-harvest seed quality assessment. Possible management practices before harvest can be used to improve total seed Ca and possibly germination. Further research will be needed to verify these results under both irrigated and non-irrigated conditions and to determine the earliest possible sampling time for accurate estimation of seed Ca concentrations.

Table 1. Analysis of variance for effects of cultivar, maturity class, sampling time, Ca treatment, and their interactions on peanut seed weight, seed Ca concentration, and total seed Ca.

	P value†			
Source of variation	Seed	Seed Ca	Total	
	weight	concentration	seed Ca	
Cultivar	< 0.01	< 0.01	0.14	
Maturity class	< 0.01	< 0.01	< 0.01	
Sampling time	< 0.01	< 0.01	< 0.01	
Ca treatment	< 0.01	< 0.01	< 0.01	
Cultivar × Maturity class	0.16	0.90	0.80	
Cultivar × Sampling time	0.65	0.53	0.66	
Cultivar $\times$ Ca treatment	0.46	0.42	0.70	
Maturity class × Sampling time	< 0.01	< 0.01	< 0.01	
Maturity class × Ca treatment	0.54	0.03	< 0.01	
Sampling time × Ca treatment	0.18	< 0.01	< 0.01	
Cultivar × Maturity class × Sampling time	0.06	0.25	0.29	
Cultivar $\times$ Maturity class $\times$ Ca treatment	0.79	0.95	0.99	
Cultivar $\times$ Sampling time $\times$ Ca treatment	0.69	0.71	0.90	
Maturity class × Sampling time × Ca treatment	0.76	0.77	0.63	
Cultivar $\times$ Maturity class $\times$ Sampling time $\times$ Ca treatment	0.77	0.99	0.96	

 $<sup>\</sup>dagger$  P values were calculated using the least square means (LSMEANS) statement of SAS with the Kenward-Roger (KR) adjustment. P values < 0.05 indicate significant effects.

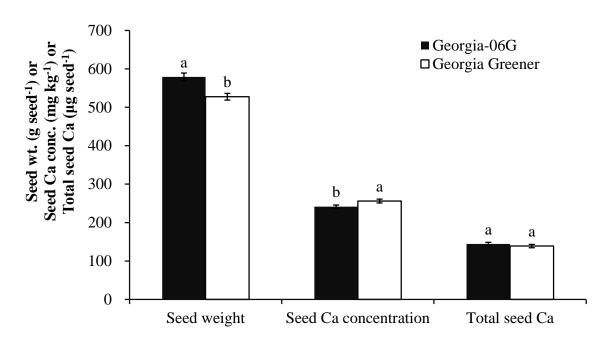


Figure 1. Peanut seed weight, seed Ca concentration, and total seed Ca in Georgia-06G and Georgia Greener. Data were combined for all Ca treatments, sampling times, and maturity classes due to lack of interactions. Means within each seed trait followed by the same letter are not significantly different at  $\alpha=0.05$ .

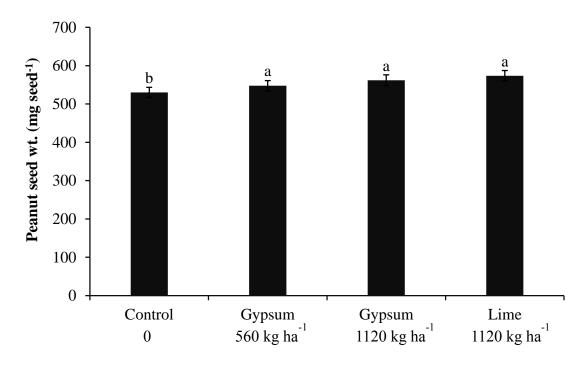


Figure 2. Seed weight of peanut grown with no Ca supplement, gypsum at 560 and 1120 kg ha<sup>-1</sup>, and lime at 1120 kg ha<sup>-1</sup>. Data were combined for Georgia-06G and Georgia Greener cultivars, sampling times (4, 2, and 0 wk prior to harvest), and maturity classes (yellow 1, yellow 2, orange, brown, and black) due to lack of interactions. Means followed by the same letter are not significantly different at  $\alpha = 0.05$ .

Table 2. Seed Ca concentration and total seed Ca of peanut grown with no Ca supplement, gypsum at 560 and 1120 kg ha<sup>-1</sup>, and lime at 1120 kg ha<sup>-1</sup> at 4, 2, and 0 wk before harvest. Data were combined for Georgia-06-G and Georgia Greener cultivars and maturity classes (yellow 1, yellow 2, orange, brown and black) due to lack of interactions. Means within each column followed by the same letter are not significantly different at  $\alpha = 0.05$ .

		Sampling time				
Ca source	Rate	4 wk before harvest	2 wk before harvest	0 wk before harvest		
		Seed Ca concentration				
	kg ha <sup>-1</sup>		mg kg <sup>-1</sup>			
Control	0	190.9 b	213.0 d	204.8 b		
Gypsum	560	200.3 b	245.9 c	210.2 b		
Gypsum	1120	246.8 a	291.3 b	300.6 a		
Lime	1120	258.8 a	344.2 a	278.2 a		
		Total seed Ca				
	kg ha <sup>-1</sup>		μg seed <sup>-1</sup>			
Control	0	85.0 c	115.5 c	127.4 b		
Gypsum	560	95.3 c	140.2 c	130.5 b		
Gypsum	1120	112.6 b	174.9 b	196.3 a		
Lime	1120	127.9 a	210.5 a	182.8 a		

Table 3. Seed Ca concentration and total seed Ca of peanut grown with no Ca supplement, gypsum at 560 and 1120 kg ha<sup>-1</sup>, and lime at 1120 kg ha<sup>-1</sup> within maturity classes by Williams and Drexler (1981), including yellow 1, yellow 2, orange, brown, and black. Data were combined for all Georgia-06G and Georgia Greener cultivars and sampling times (4, 2, and 0 wk prior to harvest) due to lack of interactions. Means within each column followed by the same letter are not significantly different at  $\alpha = 0.05$ .

Ca source	Rate	Maturity class						
		Yellow 1	Yellow 2	Orange	Brown	Black		
		Seed Ca concentration						
	kg ha <sup>-1</sup>	mg kg <sup>-1</sup>						
Control	0	209.22 b	177.22 b	181.67 c	222.93 c	223.33 b		
Gypsum	560	202.62 b	200.73 b	215.37 b	243.65 c	231.56 b		
Gypsum	1120	267.46 a	244.13 a	293.33 a	291.25 b	301.69 a		
Lime	1120	253.53 a	259.26 a	294.62 a	336.30 a	325.02 a		
		Total seed Ca						
	kg ha <sup>-1</sup>	μg seed <sup>-1</sup>						
Control	0	79.66 b	85.47 b	109.95 b	141.03 c	130.47 c		
Gypsum	560	75.41 b	95.79 b	130.66 b	163.71 c	144.38 c		
Gypsum	1120	106.84 a	122.33 a	187.67 a	202.67 b	186.87 b		
Lime	1120	101.61 a	128.06 a	190.81 a	235.32 a	212.94 a		

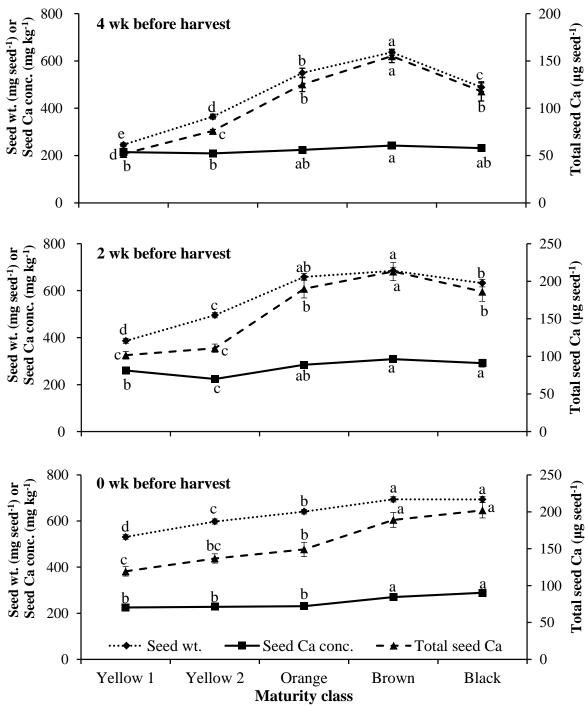


Figure 3. Seed weight, seed Ca concentration, and total seed Ca of peanut within maturity classes by Williams and Drexler (1981), including yellow 1, yellow 2, orange, brown, and black at 4, 2, and 0 wk before harvest. Data were combined for all Georgia-06G and Georgia Greener cultivars and Ca treatments (0,560, and 1120 kg ha<sup>-1</sup> gypsum and 1120 kg ha<sup>-1</sup> lime) due to lack of interactions. Means within each seed trait followed by the same letter are not significantly different at  $\alpha = 0.05$ .

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# III. Effect of Ca Source and Timing on Runner Peanut Production

#### Abstract

Peanuts require sufficient Ca in the pegging zone for proper development. Many Ca supplements are available to peanut producers; however, the efficacy of these products is not well understood. The objective of this study was to evaluate the effectiveness of various Ca sources and timing of their applications on peanut yield, grade, seed Ca, and germination. This study was conducted under non-irrigated and irrigated conditions at the Wiregrass Research and Extension Center (WREC) in Headland, AL, in 2012 and 2013, and under non-irrigated conditions at the Gulf Coast Research and Extension Center (GCREC) in Fairhope, AL, in 2013. Three commercial types of gypsum, AgriCal, which is a flue gas desulfurized (FGD) gypsum; USG 500, which is a naturally mined gypsum; and PCS Wetbulk, which is a phosphogypsum; agricultural lime; and Hi-Cal, which is a liquid Ca fertilizer derived from calcium chloride, were compared as sources of Ca. Results showed that effectiveness of the three types of gypsum were not significantly different when applied at the same rate, but USG 500 tends to perform better than AgriCal and PCS Wetbulk. Lime and gypsum were found to be equal source of Ca. Under irrigated conditions, Hi-Cal did not significantly improve peanut yield, grade, or seed quality compared to the untreated control and the dry forms of gypsum and lime. Timing of Ca treatments included application of 1120 kg ha<sup>-1</sup> gypsum at planting and early bloom, split application of gypsum at planting/early bloom and early/mid bloom, combined application of gypsum at early bloom and Hi-Cal at mid bloom, and lime at planting and gypsum at early bloom. Results showed that application of gypsum at planting and early bloom were equally effective at all locations in 2013. Split application of 1120 kg ha<sup>-1</sup> gypsum equally at early/mid

bloom tends to have superior performance compared to a single application at early bloom with an equivalent rate. Combined application of gypsum and lime did not significantly improve peanut yield and grade compare to plots receiving only lime at planting. Yield, grade, seed Ca, and germination did not increase when Hi-Cal was applied at mid bloom as a supplement to gypsum applied at early bloom.

## Introduction

Calcium is required for proper peanut pod development. Unlike many nutrients, Ca in the pod is obtained directly from the soil rather than from the root or shoot system (Wiersum, 1951; Skelton and Shear, 1971; Kiesling and Walker, 1982). Calcium must be present in the pegging zone, which is the top 7 to 9 cm of the soil where peanuts develop. Peanuts are most commonly grown in sandy surface soils because it is conducive to digging, which is required for harvest. Sandy surface soils, especially in the major peanut producing region of the southeastern United States, are known for their low ability to retain cations such as Ca. Peanut producers usually increase pegging zone Ca by applying Ca supplements, such as gypsum, lime, and liquid Ca fertilizers.

Lime and gypsum have been shown to improve peanut yield, grade, and germination (Hartzog and Adams, 1973; Hallock and Allison, 1980a). While gypsum does not increase soil pH as lime does, gypsum is more soluble than lime. Increased solubility enhances diffusion of Ca to peanut. However, availability of Mg and K can be negatively affected following gypsum application due to competition for uptake among these cations (Alva and Gascho, 1991). The higher solubility of gypsum makes it more susceptible to leaching than lime after heavy rainfall. Liquid Ca fertilizers are marketed as foliar sprays or direct soil applications with or without

Liquid Ca fertilizers are readily available to developing peanut pods. However, the highly soluble forms of liquid Ca fertilizers have great leaching potential. This, combined with lower total Ca content in liquid Ca fertilizers compared to dry forms of gypsum and lime makes the actual effectiveness of liquid Ca fertilizers questionable. A few studies have shown that liquid Ca fertilizers were not as effective as dry forms of gypsum and lime (Hartzog and Adams, 1973; Hallock, 1975; Walker, 1975; Adams and Hartzog, 1979).

Various Ca supplements are currently available to peanut producers. Naturally mined gypsum, also known as landplaster, has been widely used by peanut producers to elevate soil Ca in the pegging zone. In recent years, synthetic gypsum, such as flue gas desulfurized (FGD) gypsum and phosphogypsum, have also been used. Previous studies reporting effectiveness of different Ca sources have had inconsistent conclusions due to differences in background Ca level, rainfall pattern, peanut cultivars, and particle size of different materials (Hartzog and Adams, 1973; Daughtry and Cox, 1974; Sullivan et al., 1974; Hallock, 1975; Walker, 1975; Adams and Hartzog, 1979; Gascho et al., 1993). The compositions of currently available liquid Ca fertilizers are varied, for example, Hi-Cal is derived from calcium chloride and TigerCal 30<sup>TM</sup> is derived from chelated calcium carbonate. Manufacturers claim these liquid products are effective on improving peanut yield and grade, but there is no published literature that have evaluated effectiveness of them on cultivated peanut varieties in the Southeast.

Timing of Ca fertilizer application to obtain the optimum yield and grade is also important for peanut producers. Lime is usually applied at planting, since it stays in the pegging zone for a relatively long time. A single application of gypsum at early bloom is a currently common practice among peanut producers. A few studies have been performed to evaluate timing of Ca

fertilizer application, but the results are varied (Reed and Brady, 1948; Hallock, 1975; Hallock and Allison, 1980a). Furthermore, the efficacy of split applications of gypsum is unknown. Therefore, the objective of this study is to evaluate effect of different Ca sources and timing of their application on peanut yield, grade, seed Ca, and germination.

### **Materials and Methods**

Field experiments were conducted at the Wiregrass Research and Extension Center (WREC) in Headland, AL (31.36 °N, 85.32 °W), under both non-irrigated and irrigated conditions in 2012 and 2013, and at the Gulf Coast Research and Extension Center (GCREC) in Fairhope, AL (30.55 °N, 87.87 °W), under non-irrigated condition in 2013. This study was performed on three soil series in Alabama, including Dothan sandy loam (fine-loamy, kaolinitic, thermic Plinthic Kandiudults) and Lucy loamy sand (loamy, kaolinitic, thermic Arenic Kandiudults) at WREC, and Malbis fine sandy loam (fine-loamy, siliceous, subactive, thermic Plinthic Paleudults) at GCREC. At each location, the plots (2 to 4 m width and 8 to 10 m length) were organized as a split-plot design with 4 replications. The main plot factor was Ca source or timing of application, while the sub-plot factor was peanut cultivar. Two runner-type peanut cultivars, relatively large-seeded Georgia-06G and medium-seeded Georgia Greener, were selected for evaluation at WREC due to their size difference and popularity in the Southeast. Georgia-06G and a new cultivar, medium-seeded Georgia-09B, were compared at GCREC. Peanut production in each test followed typical management practices in the area, including herbicide and fungicide regimes, but no fertilizers were applied other than the Ca treatments. Rainfall data in Headland and Fairhope were collected by Alabama Mesonet at the weather stations (Agricultural Weather

Information Service, Inc., Auburn, AL) located within 2 km from each test at WREC and GCREC.

For the Ca source trial, three commercial types of gypsum, including AgriCal® (FGD gypsum), PCS Wetbulk<sup>®</sup> (phosphogypsum), and USG 500<sup>®</sup> (mined gypsum), were used. In addition, two locally available liming materials were used, including calcitic lime that was used at WREC (Tri-State Lime, LLC, Arlington, GA) and dolomitic lime used at GCREC (Farmers Favorite Fertilizer Company, Moultrie, GA). These sources were investigated under both nonirrigated and irrigated conditions at WREC and non-irrigated conditions at GCREC. Lime was applied at planting at a rate of 1120 kg ha<sup>-1</sup>, whereas gypsum was applied at early bloom (approx. 40 to 50 days after planting, DAP) at a rate of 560 kg ha<sup>-1</sup> in 2012 and 1120 kg ha<sup>-1</sup> in 2013. Each Ca source was broadcasted by hand with gypsum or lime concentrated over the peanut rows. In 2012, two liquid Ca fertilizer applications, Hi-Cal® at a rate of 374 L ha<sup>-1</sup> and TigerCal 30<sup>TM</sup> at a rate of 94 L ha<sup>-1</sup> at early bloom were also compared. In 2013, only Hi-Cal<sup>®</sup> applied at the same rate as 2012 was evaluated. The liquid Ca fertilizers were diluted in water according to manufacturer's instructions and applied using a field sprayer to simulate application through irrigation. Since there was no irrigation facility at GCREC, liquid Ca fertilizers were only evaluated under irrigated conditions at WREC. Irrigation was applied at 2 to 3 mm following application to negate confounding effects of additional water applied to these treatments.

The Ca timing study was first performed under irrigated conditions at WREC in 2012, including two split applications of AgriCal (560 and 1120 kg ha<sup>-1</sup> split into two equal applications at early/mid bloom), a late application of AgriCal (560 kg ha<sup>-1</sup> at late bloom), and a split application of AgriCal with Hi-Cal (560 kg ha<sup>-1</sup> AgriCal at early bloom and 37.4 L ha<sup>-1</sup> Hi-

Cal at mid bloom). In 2013, the Ca timing study was modified. Two split applications of AgriCal (1120 kg ha<sup>-1</sup> split into two equal applications at planting/early bloom and at early/mid bloom), an early application of AgriCal (1120 kg ha<sup>-1</sup> at planting), a split application of lime with AgriCal (1120 kg ha<sup>-1</sup> lime at planting and 560 kg ha<sup>-1</sup> AgriCal at early bloom) were compared under both irrigated and non-irrigated conditions at WREC and GCREC. In addition, a split application of AgriCal with Hi-Cal (1120 kg ha<sup>-1</sup> AgriCal at early bloom and 37.4 L ha<sup>-1</sup> Hi-Cal at mid bloom) was evaluated under irrigated conditions at WREC.

Pegging zone soil samples (0 to 7 cm depth) were taken at planting, mid bloom (approx. 70 to 80 DAP), and harvest. Fourteen cores of soil were collected from each sub-plot with a handheld soil probe and mixed well in a bucket to provide composite samples. Soils were then dried at 60 °C for 48 h and ground using a mortar and pestle to pass a 2-mm sieve. Soil pH was determined according to a method by Hue and Evans (1986). Briefly, 10 g soil sample was mixed with 20 mL deionized water (D.I. water) and shaken for 10 min. After settling 30 min, pH was measured using a pH meter (Sevencompact<sup>TM</sup> pH/Ion meter S220, Mettler-Toledo LLC, Columbus, OH). Soil Ca, Mg, and K concentrations were determined using the Mehlich 1 extraction (Mehlich, 1953). In detail, 5 g soil sample was shaken with 20 mL Mehlich 1 extractant solution for 5 min, centrifuged at 1000 rcf (IEC Model K centrifuge, International Equipment Company, Needham Heights, MA) for 10 min and then filtered with Whatman No. 40 filter paper (GE Healthcare Life Sciences, Pittsburgh, PA). The filtrate was then analyzed with inductively coupled plasma (ICP) spectroscopy (Spectro Ciros CCD, side on plasma, SPECTRO Analytical Instruments Inc., Kleve, Germany).

Peanuts were planted on Jun. 7 and harvested on Oct. 31 in 2012 and 2013 at WREC, and were planted on May. 17 and harvested on Oct. 10 in 2013 at GCREC. Due to a harvesting

problem in 2012, we were unable to recover peanut yield, grade, seed Ca or germination data for Ca source and timing trials performed at WREC under irrigated conditions.

Peanut yield was determined after drying to approximately 10% moisture. Peanut grade was calculated as the percentage of sound mature kernels (SMK) in a 250 g sample. The SMK were then dried at 60 °C for 48 h and finely ground in a coffee grinder (Hamilton Beach Inc., Picton, Canada). Seed Ca was determined by microwave digestion using a Mars Xpress microwave (CEM Corp., Matthews, NC) using the modified EPA 3051A procedure (USEPA, 2007) followed by Ca determination by ICP. In detail, 1 g finely ground peanut sample was predigested overnight in 10 mL 30% (v/v) hydrogen peroxide and then microwave digested for 15 min in 10 mL concentrated nitric acid. The final digested material was filtered using Whatman No. 40 filter paper, and the volume was adjusted to 100 mL with D.I. water before analysis by ICP.

Warm and cold germination tests were performed by the Georgia Department of Agriculture Seed Laboratory in Tifton, GA, according to procedures defined by the Association of Official Seed Analysis (AOSA). One hundred and 200 seeds were used for warm and cold germination, respectively. Germination percentage was determined by treating seed with the fungicide Vitavax<sup>®</sup> PC (Bayer CropScience LP, Research Triangle Park, NC) and placing on germination paper (25.4 × 38.1 cm). Seeds were then placed in a germinator at 25 ℃ for warm germination test and at 10 ℃ for cold germination test. After 10 days in the germinator, the percentage of germination was recorded.

Characteristics of Ca sources were also analyzed. Solubility in diluted salt solution was determined. In detail, 0.5 g gypsum or lime sample was mixed with 50 mL 0.05 M NaCl solution and shaken for 2h. The suspension was filtered with Whatman No. 40 filter paper. The

filtrate was analyzed for Ca by ICP and the solubility was calculated. For total Ca, K, and Mg content analysis, 0.1 g gypsum or lime was acid-digested using the modified EPA 3051A procedure described above following analysis on ICP (USEPA, 2007). Particle size was determined by passing 500 g gypsum or lime through 2- and 1-mm sieves, and the percentage of particles with diameters > 2 mm, between 1 and 2 mm, and < 1 mm was calculated on weight basis. Three analytical replications were performed for each test.

Mixed models methodology as implemented in SAS PROC MIXED was used to analyze the data (Littell et al., 2006). Calcium source and timing treatment and peanut variety were fixed effects. Block was treated as the only random effect. To test for differences among Ca source and timing treatments, the Kenward-Roger (KR) adjustment was applied to the least square means (LSMEANS) statement of SAS software.

## **Results and Discussion**

# Growth conditions for each test

Soil pH was between 5.5 and 6.5 for each site/year. All sites had initial soil calcium levels similar to or greater than 150 mg kg<sup>-1</sup>, which is at or above the critical value established for traditional runner-type peanuts in Alabama (Hartzog and Adams, 1976). In both 2012 and 2013, the irrigated trial at WREC (Dothan sandy loam) had higher initial soil Ca and K level but lower soil Mg level than the non-irrigated trial (Lucy loamy sand). Due to continuous liming practices with dolomitic lime, initial soil Ca and Mg levels at GCREC were notably higher than at WREC. The Ca:K ratio in all the soils evaluated was above the 3:1 ratio recommended for peanut production by University of Georgia (Kissel and Sonon, 2008; Table 4). The 30 yr mean rainfall

during the peanut growing season (May through October) was 675 and 855 mm at the Headland and Fairhope weather stations, respectively. In 2013, rainfall at both stations was 30-40% higher than the average; therefore, the 2013 growing season was considered as a wet year (Table 4).

# Ca source trial

Source and rate significantly affected peanut yield (P = 0.042) under non-irrigated conditions at WREC in 2012 (Figure 4). A significant treatment × cultivar interaction (P = 0.004) was noted on peanut yield; therefore, yield data was interpreted by cultivar. Yield of Georgia Greener was not significantly improved with addition of Ca supplements compared to the untreated control; however, USG 500 produced a significantly higher yield than PCS Wetbulk (Figure 4A). Georgia-06G showed greater yield response to different Ca treatments. When applied at a rate of 560 kg ha<sup>-1</sup> at early bloom, AgriCal and USG 500 notably increased yield of Georgia-06G compared to the untreated control, whereas PCS Wetbulk did not, but there was no difference among the three commercial types of gypsum. Application of 1120 kg ha<sup>-1</sup> AgriCal at early bloom or lime at planting significantly improved yield compared to the control treatment (Figure 4B).

In 2013, no significant treatment × cultivar interaction (P > 0.5) was noted on peanut yield for each tests; therefore, data were combined for all cultivars. There was no significant yield response to Ca source and rate at all the locations (Table 5). Lack of effect of gypsum and lime applications in all the three trials in 2013 suggests that Ca was not the limiting factor for peanut production. At WREC, average peanut yield under irrigated conditions was 30% higher than non-irrigated conditions (Table 5), suggesting that peanut yield can be increased with regular irrigation even in a year which received above-normal rainfall (e.g., 2013). Rainfall can be both

beneficial and detrimental to Ca availability. Rainfall solubilizes Ca and improves its diffusion toward the developing pod; however, excessive rainfall can cause leaching and reduced Ca availability. Although rainfall was high in 2013, rainfall events were likely not intense enough to completely leach Ca from the pegging zone, but frequent enough to maintain adequate Ca availability even when soil Ca levels were near critical values. In the non-irrigated study conducted in 2012, rainfall was more limiting, which likely resulted in greater periods of Ca deficiency. Studies by Cox et al. (1976) and Howe et al. (2012) also indicate that adequate moisture is important for Ca availability to peanut.

There was no significant treatment  $\times$  cultivar interaction effect (P > 0.2) on peanut grade, seed Ca concentration, or germination in all the four trials in 2012 and 2013; therefore, data were combined for all cultivars at each site-year.

Under non-irrigated conditions in 2012, application of 560 kg ha<sup>-1</sup> USG 500 at early bloom significantly increased peanut grade compared to the untreated control, whereas AgriCal and PCS Wetbulk did not when applied at the same rate (Table 6). Application of lime at planting or gypsum at early bloom at 1120 kg ha<sup>-1</sup> showed equal efficacy on improving peanut grade. In 2013, peanut grade response to additional Ca was only observed at WREC under non-irrigated conditions. When applied at 1120 kg ha<sup>-1</sup> at early bloom, USG 500 and AgriCal significantly increased peanut grade compared to untreated control, whereas PCS wetbulk did not; however, effectiveness of the three types of gypsum was not significantly different. In both 2012 and 2013, lime and gypsum showed similar efficacy on improving peanut grade. Irrigation did not increased peanut grade at WREC in 2013 (Table 6). These results are consistent with results for yield; although there were a few small differences in grade in the non-irrigated trial at WREC in 2013 than were noted for yield.

In 2012, Ca supplementation significantly improved seed Ca concentration, but no significant difference among Ca sources and rates was noted (Table 7). In 2013, seed Ca showed a notable response to added Ca at WREC but not at GCREC. Under both irrigated and non-irrigated conditions, USG 500 significantly increased seed Ca concentration above the untreated control, whereas AgriCal did not; however, efficacy was not significantly different among the three types of gypsum. Lime showed significantly higher efficacy on increasing seed Ca concentration than gypsum compared to non-irrigated conditions in 2013 (Table 7).

Numerically, the highest seed Ca concentration in the current study occurred following application of 1120 kg ha<sup>-1</sup> lime at planting in all the four site-years.

Under irrigated conditions, Hi-Cal did not significantly increase seed Ca concentration above the untreated control or dry forms of gypsum and lime. Averaged seed Ca concentration increased from 308 mg kg<sup>-1</sup> under non-irrigated conditions to 366 mg kg<sup>-1</sup> under irrigated conditions, indicating seed Ca concentration may be increased with irrigation. Seed Ca concentration in peanuts grown at GCREC was 2.5-fold higher than WREC due to the dramatically high soil Ca level combined with high rainfall at GCREC (Table 7).

Lime showed equal effectiveness as gypsum on improving peanut yield, grade, and seed quality in all the four site-years. In 2012, neither type of gypsum could significantly improve warm and cold germination compared to the untreated control; however, USG 500 treated peanuts tended to produce higher warm and cold germination rates than AgriCal and PCS Wetbulk when applied at 560 kg ha<sup>-1</sup> (Table 8). In 2013, Ca treatments significantly influenced warm and cold germination at WREC but not at GCREC. Under non-irrigated conditions, addition of USG 500 and AgriCal significantly increased warm and cold germination compared to the untreated control, whereas PCS wetbulk did not; however, there was no significant

difference among the three types of gypsum. Under irrigated conditions, Ca supplementation significantly increased warm and cold germination; however, the efficacy among different Ca sources and rates was not significantly different (Table 8).

Maximum germination, defined as >95% germination, for traditional runner-type peanuts (e.g., Florunner and Southern Runner) occurred when seed Ca concentration reached 400 to 420 mg kg<sup>-1</sup> (Adams et al., 1993; Cox et al., 1982). However, work by Howe et al. (2012) found that currently cultivated peanuts, namely Georgia-06G and Georgia Green, required 600 mg kg<sup>-1</sup> seed Ca to achieve maximum germination. In the current study, maximum germination occurred only at GCREC, and seed Ca was > 800 mg kg<sup>-1</sup>. While there was no significant difference between the two cultivars used at GCREC, there may be differences when compared to previous cultivars. Tillman et al. (2010) suggested that varied response in seed Ca concentration and germination to Ca could be attributed to the genetic variation of different cultivars.

The current study found that the three commercial types of gypsum, AgriCal, USG 500, and PCS Wetbulk had similar effectiveness on improving peanut yield, grade, and seed quality (e.g., seed Ca concentration and germination). A few studies that have compared different Ca sources had similar conclusions. Daughtry and Cox (1974) reported that no significant difference on peanut yield was noted among phosphogypsum, finely-ground landplaster, and granular landplaster. Work by Grichar et al. (2004) found that FGD gypsum and "agricultural gypsum" were equally effective sources of supplemental Ca for peanuts.

Although overall effectiveness of the gypsum sources were similar, USG 500, which is a naturally mined type of gypsum, tended to perform consistently the best and PCS Wetbulk, which is a phosphogypsum, tended to consistently perform the worst using all the metrics of yield, grade, seed Ca concentration, and warm and cold germination. These results can be

attributed to differences in total Ca content and particle size among the three types of gypsum. The total Ca in AgriCal and USG 500 is similar and is 15% greater than in PCS Wetbulk (Table 9). Therefore, it has been found that under conditions evaluated in this study, AgriCal and USG 500 significantly improved peanut yield, grade, seed Ca and germination compared to untreated control, but PCS Wetbulk did not due to its lower total Ca input. Both AgriCal and PCS Wetbulk contains >50% fine (<1 mm) particles. In contrast, USG 500 contains only 20% fine particles but >50% coarse (> 2mm) particles (Table 9). It has been reported that the smaller, finer particles are readily dissolved, while the larger particles dissolve more slowly (Bolan et al., 1991; Alva et al., 1989). Coarse gypsum has been shown to supply Ca in the pegging zone longer than fine gypsum (Keisling and Walker, 1978). Fine particles may be readily available to peanuts in the early season, while the coarse particles persist longer and may be able to supply Ca to developing pods in the late season. The particle size of USG 500 ranges from approximately 5 to <0.1 mm. Therefore, USG 500 may have met the Ca requirements by providing Ca to peanuts throughout the growing season.

The liquid Ca fertilizer, Hi-Cal, was the least effective on supplying Ca to peanut, possibly due to the lower total amount of Ca input with application of liquid Ca fertilizers. In this study, 45 kg Ca ha<sup>-1</sup> was added to soil with 374 L ha<sup>-1</sup> Hi-Cal, which is < 10% of Ca added with 1120 kg ha<sup>-1</sup> AgriCal (250 kg Ca ha<sup>-1</sup>). Many other studies have also shown lower effectiveness of liquid Ca fertilizers relative to dry forms of gypsum and lime on peanut (Hartzog and Adams, 1973; Hallock, 1975; Walker, 1975; Adams and Hartzog, 1979).

Lime has lower Ca availability than gypsum due to its lower solubility and consequently a diffusion limitation. However, with irrigation or sufficient rainfall, the limitation of diffusion can be largely reduced. Furthermore, total Ca input from lime was approximately 50% greater

than gypsum (Table 9). Soil Ca increased from 198 mg kg<sup>-1</sup> at mid bloom to 256 mg kg<sup>-1</sup> at harvest in 2013 at WREC, when lime was applied at planting to soil with an initial Ca level of 166 mg kg<sup>-1</sup>. Lime applied at planting may be able to supply Ca to peanut throughout development and thus produce high seed Ca concentration, as long as adequate moisture is available to maintain dissolution of the lime.

# Ca timing trial

In 2013, there was no significant treatment  $\times$  cultivar interaction effect on peanut yield, grade, seed Ca, or germination (P > 0.1) for the Ca timing trial at either location; therefore, data were combined across all cultivars.

Peanut yield and grade showed no response to Ca timing treatments at all locations (Table 10 and 11). However, previous studies showed that application of gypsum at early bloom provided more available Ca (Reed and Brady, 1948) and tended to produce higher yield than application at planting (Hallock and Allison, 1980a).

Timing of Ca fertilizer applications significantly influenced seed Ca concentration and germination under both irrigated and non-irrigated conditions at WREC (Tables 12 and 13). Results showed that application of gypsum at planting and at early bloom were equally effective on improving seed Ca concentration and germination under both irrigated and non-irrigated conditions at WREC (Tables 12 and 13). Split applications of AgriCal at early (560 kg ha<sup>-1</sup>) and mid-bloom (560 kg ha<sup>-1</sup>) produced significantly higher seed Ca than the untreated control at all the locations. The same rate split at planting and early bloom did not increase seed Ca compared to the control, even though there was no significant difference between these two split application treatments, except under irrigated conditions at WREC. In this trial, the split

application at early/mid-bloom produced significantly higher seed Ca than split application at planting/early bloom (Table 12).

A lack of yield and grade response in the current study is possibly due to the above-normal rainfall in 2013. Rainfall received during the peanut growing season was 911 and 1200 mm at WREC and GCREC, respectively, which was 240 and 290 mm above normal. Infrequent but high magnitude rainfall can reduce soil Ca concentrations near the soil surface (Keisling and Walker, 1978); therefore, added Ca supplements may have been leached out of the pegging zone following a few tropical storms in 2013, and did not significantly affect peanut yield and grade. Even though seed Ca differed, differences were relatively small.

Timing of Ca application affected cold germination results more than warm germination.

Except at GCREC, application of any Ca treatment improved warm germination compared to the untreated control. Results for cold germination were similar, except there were some additional differences among timing treatments, but only in the irrigated treatments conducted at WREC.

Peanut grown with a split application of AgriCal at early/mid-bloom (560/560 kg ha<sup>-1</sup>) had significantly higher cold germination than a single application of 1120 kg ha<sup>-1</sup> at early bloom (Table 13). Few other studies have evaluated the effectiveness of split applications of gypsum.

Under irrigated conditions at WREC, a single application of Hi-Cal at early bloom did not improve seed Ca compared to the untreated control and the dry forms of gypsum and lime; however, application of 1120 kg ha<sup>-1</sup> AgriCal at early bloom, together with Hi-Cal at mid bloom as supplemental Ca, tended to produce greater seed Ca compared to plots that received only 1120 kg ha<sup>-1</sup> AgriCal at early bloom (Table 12). Hallock (1975) compared split applications of liquid Ca fertilizers with a single application and reported greater yield and gross value with split application; however, improved peanut yield and grade relative to untreated control was

observed only when gypsum and liquid Ca fertilizers were applied together. The current study reached the same conclusion, but only with respect to seed Ca concentration. Treatment receiving 1120 kg ha<sup>-1</sup> lime at planting with additional AgriCal at early bloom did not show improved efficacy on improving seed Ca concentration or germination above treatment receiving only lime at planting at all locations (Tables 12 and 13). Both warm and cold germination at WREC were notably improved by supplemental Ca, but there was no significant difference among Ca timing treatments, except for cold germination under non-irrigated conditions at WREC.

### **Conclusions**

There was no statistically significant differences among the three commercial types of gypsum (AgriCal, PCS Wetbulk, and USG 500) on improving peanut yield, grade, seed quality and soil Ca; therefore, peanut producers may use either of them to supply supplemental Ca. However, USG 500 tended to perform better than AgriCal and PCS Wetbulk, thus USG 500 is recommended if it is available in the market. When applied at the same rate and when moisture is not limiting, lime is as effective as gypsum for peanut production. However, lime has a lower solubility and consequently lower availability to peanut than gypsum. Irrigation may help reduce diffusion limitations, so either gypsum or lime can be used under irrigated conditions, and gypsum is recommended under non-irrigated conditions. The liquid Ca fertilizers evaluated in this study did not significantly improve peanut yield, grade, or seed quality indicators; therefore, a single application of liquid Ca fertilizer at early bloom should not be recommended, since it provides inadequate Ca for peanut development.

There was no statistical difference between application of gypsum at planting and at early bloom on improving peanut yield, grade, and seed quality indicators observed in this study.

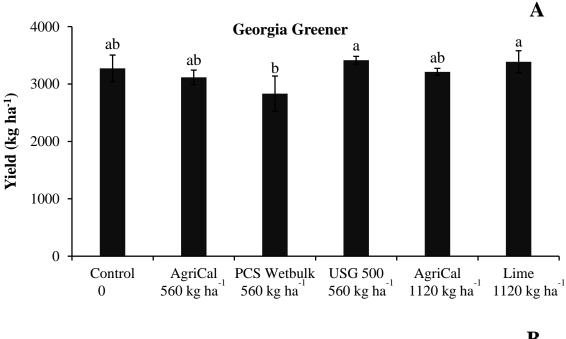
However, with more than 600 mm rainfall in July and August in 2013, applied gypsum may have been leached out of the pegging zone, thus the benefits of application at early bloom relative to planting may be offset. Seed Ca was found to be highly influenced by Ca treatment and is a good indicator of soil Ca availability. Split applications of gypsum at early/mid-bloom (560/560 kg ha<sup>-1</sup>) significantly improved seed Ca relative to the untreated control, and this treatment tends to have greater effectiveness on improving seed Ca relative to split applications at planting/early bloom. Therefore, split applications of gypsum at early/mid bloom are recommended for peanut producers. Since combined application of lime applied at planting and additional gypsum at early bloom did not show benefit over plots that received only lime at planting, these data suggest that extra gypsum at blooming may not be necessary if lime has been applied at planting. These conclusions were based on one year's data at different locations; therefore further studies will be needed to verify these results.

Table 4. Characteristics of each field trial conducted in 2012 and 2013 including location, irrigation status, soil type, pH, initial soil Ca, Mg, and K concentration in the pegging zone, and rainfall during the peanut growing season (May-October) of each study.

Year	Location	Nearest City	Irrigation	Soil:	ъU	Init	Initial soil nutrients		Rainfall
1 ear	name†	Nearest City	Irrigation	S011.	pН	Ca	K	Mg	Kailliali
							mg kg <sup>-1</sup>		mm
2012	WREC	Headland, AL	No	Lucy ls	5.6	164	22	23	566
2012	WREC	Headland, AL	Yes	Dothan sl	5.9	276	45	22	566
2013	WREC	Headland, AL	No	Lucy ls	6.1	148	15	18	911
2013	WREC	Headland, AL	Yes	Dothan sl	5.8	191	22	14	911
2013	GCREC	Fairhope, AL	No	Malbis sl	5.7	513	55	127	1200

<sup>†</sup> Location name abbreviations: WREC = Wiregrass Research and Extension Center; GCREC = Gulf Coast Research and Extension Center.

<sup>‡</sup> Soil descriptions: Dothan Fine-loamy, kaolinitic, thermic Plinthic Kandiudults; Lucy Loamy, kaolinitic, thermic Arenic Kandiudults; Malbis Fine-loamy, siliceous, subactive, thermic Plinthic Paleudults; sl = sandy loam and ls = loamy sand.



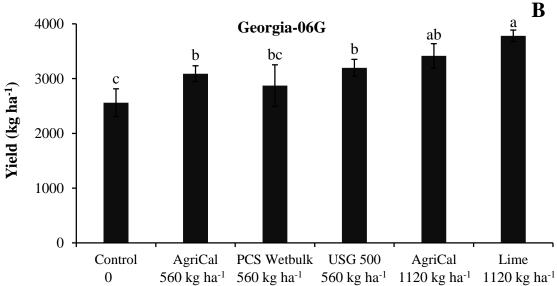


Figure 4. Peanut yield for Ca source trial under non-irrigated conditions at Wiregrass Research and Extension Center (WREC) in 2012. Data was interpreted considering the two peanut cultivars, Georgia-06G and Georgia Greener, separately, because there was a significant cultivar  $\times$  treatment interaction. Means followed by the same letter within a graph are not significantly different at  $\alpha=0.05$ .

Table 5. Peanut yield at Wiregrass Research and Extension Center (WREC) and Gulf Coast Research and Extension Center (GCREC) in 2013. Data were combined for Georgia-06G and Georgia Greener cultivars at WREC and Georgia-06G and Georgia-09B cultivars at GCREC due to lack of cultivar  $\times$  treatment interaction. Means within each column followed by the same letter are not significantly different at  $\alpha=0.05$ .

			Location	
Ca source	Rate	GCREC	WREC	WREC
		Non-irrigated	Non-irrigated	Irrigated
			Yield	
	kg or L ha <sup>-1</sup>		kg ha <sup>-1</sup>	
Control	0	3790 a	2320 a	3140 a
AgriCal	560	4140 a	2360 a	2940 a
AgriCal	1120	4040 a	2130 a	3090 a
PCS Wetbulk	1120	3870 a	2240 a	2990 a
USG 500	1120	3420 a	2340 a	2930 a
Lime	1120	3500 a	2250 a	2980 a
Hi-Cal	374			2800 a

Table 6. Peanut grade represented as percentage of sound mature kernels (SMK) at Wiregrass Research and Extension Center (WREC) and Gulf Coast Research and Extension Center (GCREC) in 2012 and 2013. Data were combined for Georgia-06G and Georgia Greener cultivars at WREC and Georgia-06G and Georgia-09B cultivars at GCREC due to lack of cultivar  $\times$  treatment interaction. Means within each column followed by the same letter are not significantly different at  $\alpha = 0.05$ .

-	2	012	2013					
Ca source	Rate	WREC Non-irrigated	Rate	GCREC Non-irrigated	WREC Non-irrigated	WREC Irrigated		
	kg or L ha <sup>-1</sup>	SMK %	kg or L ha <sup>-1</sup>	SMK %				
Control	0	61.0 b	0	65.9 a	72.6 b	68.8 a		
AgriCal	560	63.4 b	1120	65.2 a	74.7 a	72.4 a		
PCS Wetbulk	560	63.0 b	1120	66.2 a	74.1 ab	71.9 a		
USG 500	560	68.0 a	1120	65.0 a	75.0 a	71.2 a		
Lime	1120	68.2 a	1120	67.2 a	73.7 ab	67.3 a		
AgriCal	1120	70.4 a	560	64.0 a	73.6 b	71.6 a		
Hi-Cal	374		374			72.6 a		

Table 7. Peanut seed Ca concentration at Wiregrass Research and Extension Center (WREC) and Gulf Coast Research and Extension Center (GCREC) in 2012 and 2013. Data were combined for Georgia-06G and Georgia Greener cultivars at WREC and Georgia-06G and Georgia-09B cultivars at GCREC due to lack of cultivar  $\times$  treatment interaction. Means within each column followed by the same letter are not significantly different at  $\alpha = 0.05$ .

	2012		2013					
Ca source	Rate	WREC Non-irrigated	Rate	GCREC Non-irrigated	WREC Non-irrigated	WREC Irrigated		
	kg or L ha <sup>-1</sup>	Seed Ca mg kg <sup>-1</sup>	kg or L ha <sup>-1</sup>		Seed Ca mg kg <sup>-1</sup>			
Control	0	253 b	0	837 a	251 c	291 b		
AgriCal	560	326 a	1120	871 a	301 bc	397 a		
PCS Wetbulk	560	339 a	1120	849 a	280 bc	352 ab		
USG 500	560	330 a	1120	853 a	331 b	407 a		
Lime	1120	375 a	1120	884 a	401 a	409 a		
AgriCal	1120	334 a	560	817 a	283 bc	367 a		
Hi-Cal	374		374			337 ab		

Table 8. Peanut warm and cold germination at Wiregrass Research and Extension Center (WREC) and Gulf Coast Research and Extension Center (GCREC) in 2012 and 2013. Data were combined for Georgia-06G and Georgia Greener cultivars at WREC and Georgia-06G and Georgia-09B cultivars at GCREC due to lack of cultivar  $\times$  treatment interaction. Means within each column followed by the same letter are not significantly different at  $\alpha = 0.05$ .

		2012		2	2013	
Ca source	Rate	WREC Non-irrigated	Rate	GCREC Non-irrigated	WREC Non-irrigated	WREC Irrigated
	kg or L ha <sup>-1</sup>	Warm germination %	kg or L ha <sup>-1</sup>	·	Varm germination	
Control	0	20.5 b	0	94.8 a	62.1 b	62.1 b
AgriCal	560	27.0 b	1120	95.0 a	75.6 a	76.1 a
PCS Wetbulk	560	29.4 b	1120	96.9 a	70.0 ab	77.3 a
USG 500	560	38.3 ab	1120	96.0 a	77.1 a	77.9 a
Lime	1120	43.0 a	1120	96.0 a	76.4 a	80.5 a
AgriCal	1120	36.1 ab	560	95.0 a	65.4 b	80.0 a
Hi-Cal	374		374			76.4 a
	kg or L ha <sup>-1</sup>	Cold germination %	kg or L ha <sup>-1</sup>		Cold germination	
Control	0	16.4 b	0	96.9 a	52.5 b	56.9 b
AgriCal	560	22.1 b	1120	95.5 a	66.8 a	68.1 a
PCS Wetbulk	560	25.8 ab	1120	94.0 a	63.4 ab	67.6 a
USG 500	560	31.8 ab	1120	94.5 a	68.8 a	72.8 a
Lime	1120	35.8 a	1120	95.9 a	67.6 a	70.3 a
AgriCal	1120	32.8 ab	560	95.9 a	56.9 b	69.9 a
Hi-Cal	374		374			66.9 a

Table 9. Characteristics of AgriCal, USG 500, PCS Wetbulk, and lime evaluated including solubility in diluted salt solution, total Ca, K, and Mg content, and particle size distribution

Ca source	Solubility in	To	Total nutrients			Particle size distribution		
Ca source	0.05 M NaCl	Ca	K	Mg	> 2 mm	1-2 mm	< 1 mm	
	g L-1	g kg <sup>-1</sup>	g kg <sup>-1</sup> mg kg <sup>-1</sup>		0/0-			
AgriCal	2.56	223.7	262.3	582.3	23.1	7.5	69.4	
PCS Wetbulk	2.47	194.1	507.4	261.3	31.6	12.3	56.1	
USG 500	2.39	227.6	244.6	391.7	56.2	23.7	20.0	
Calcitic lime	0.01	339.6	681.8	2969.6	19.2	30.1	50.7	
Dolomitic lime	0.02	390.4	551.4	6747.9	71.7	27.9	0.4	

Table 10. Peanut yield at Wiregrass Research and Extension Center (WREC) and Gulf Coast Research and Extension Center (GCREC) in 2013. Data were combined for Georgia-06G and Georgia Greener cultivars at WREC and Georgia-06G and Georgia-09B cultivars at GCREC due to lack of cultivar  $\times$  treatment interaction. Means within each column followed by the same letter are not significantly different at  $\alpha=0.05$ .

				Location	
Ca source	Rate	Timing	GCREC Non-	WREC Non-	WREC Irrigated
	kg or L ha <sup>-1</sup>		irrigated 	irrigated Yield kg ha <sup>-1</sup>	
Control	0		3790 a	2320 a	3140 a
AgriCal	1120	Early bloom	4040 a	2130 a	3190 a
AgriCal	1120	Planting	3510 a	2030 a	3000 a
AgriCal	560/560	Planting/Early bloom	3680 a	2510 a	3240 a
AgriCal	560/560	Early/Mid bloom	3700 a	2460 a	2970 a
Lime	1120	Planting	3500 a	2250 a	2980 a
Lime/AgriCal	1120/560	Planting/Early bloom	3630 a	2450 a	3390 a
Hi-Cal	374	Early bloom			2800 a
AgriCal/Hi-Cal	1120/37	Early/Mid bloom			2680 a

Table 11. Peanut grade represented as percentage of sound mature kernels (SMK) at Wiregrass Research and Extension Center (WREC) and Gulf Coast Research and Extension Center (GCREC) in 2013. Data were combined for Georgia-06G and Georgia Greener cultivars at WREC and Georgia-06G and Georgia-09B cultivars at GCREC due to lack of cultivar  $\times$  treatment interaction. Means within each column followed by the same letter are not significantly different at  $\alpha=0.05$ .

				Location	
Ca source	Rate	Timing	GCREC Non- irrigated	WREC Non- irrigated	WREC Irrigated
	kg or L ha <sup>-1</sup>			SMK %	
Control	0		65.9 a	72.6 a	68.8 a
AgriCal	1120	Early bloom	65.2 a	73.6 a	72.4 a
AgriCal	1120	Planting	63.8 a	73.5 a	69.9 a
AgriCal	560/560	Planting/Early bloom	63.8 a	73.3 a	72.5 a
AgriCal	560/560	Early/Mid bloom	64.4 a	73.4 a	71.7 a
Lime	1120	Planting	67.2 a	73.7 a	67.3 a
Lime/AgriCal	1120/560	Planting/Early bloom	65.3 a	74.6 a	70.7 a
Hi-Cal	374	Early bloom			72.6 a
AgriCal/Hi-Cal	1120/37	Early/Mid bloom			71.8 a

Table 12. Peanut seed Ca concentration at Wiregrass Research and Extension Center (WREC) and Gulf Coast Research and Extension Center (GCREC) in 2013. Data were combined for Georgia-06G and Georgia Greener cultivars at WREC and Georgia-06G and Georgia-09B cultivars at GCREC due to lack of cultivar  $\times$  treatment interaction. Means within each column followed by the same letter are not significantly different at  $\alpha = 0.05$ .

				Location	
Ca source	Rate	Timing	GCREC Non- irrigated	WREC Non- irrigated	WREC Irrigated
	kg or L ha			Seed Ca mg kg <sup>-1</sup>	
Control	0		837 b	251 c	291 b
AgriCal	1120	Early bloom	871 ab	301 bc	352 b
AgriCal	1120	Planting	824 b	315 bc	382 ab
AgriCal	560/560	Planting/Early bloom	923 ab	306 bc	349 b
AgriCal	560/560	Early/Mid bloom	959 a	330 b	450 a
Lime	1120	Planting	884 ab	401 a	409 ab
Lime/AgriCal	1120/560	Planting/Early bloom	891 ab	343 ab	410 ab
Hi-Cal	374	Early bloom			337 b
AgriCal/Hi-Cal	1120/37	Early/Mid bloom			409 ab

Table 13. Peanut warm and cold germination at Wiregrass Research and Extension Center (WREC) and Gulf Coast Research and Extension Center (GCREC) in 2013. Data were combined for Georgia-06G and Georgia Greener cultivars at WREC and Georgia-06G and Georgia-09B cultivars at GCREC due to lack of cultivar  $\times$  treatment interaction. Means within each column followed by the same letter are not significantly different at  $\alpha=0.05$ .

				Location	
Ca source	Rate	Timing	GCREC	WREC	WREC
Cu Bource	Rute	1 mining	Non-	Non-	Irrigate
			irrigated	irrigated	d
			Wai	rm germinati	
	kg or L ha <sup>-1</sup>			······%	
Control	0		94.8 a	62.1 b	62.1 b
AgriCal	1120	Early bloom	95.0 a	75.6 a	76.1 a
AgriCal	1120	Planting	93.4 a	74.0 a	79.4 a
AgriCal	560/560	Planting/Early bloom	95.9 a	75.0 a	76.0 a
AgriCal	560/560	Early/Mid bloom	95.0 a	78.1 a	81.1 a
Lime	1120	Planting	96.0 a	76.4 a	80.5 a
Lime/AgriCal	1120/560	Planting/Early bloom	95.5 a	74.5 a	82.9 a
Hi-Cal	374	Early bloom			76.4 a
AgriCal/Hi-Cal	1120/37	Early/Mid bloom			77.8 a
			Col	ld germinatio	n
	kg or L ha <sup>-1</sup>			······%	
Control	0		96.6 a	52.5 b	56.9 c
AgriCal	1120	Early bloom	95.5 a	66.8 a	68.1 b
AgriCal	1120	Planting	95.7 a	65.1 a	72.5 ab
AgriCal	560/560	Planting/Early bloom	94.4 a	64.8 a	70.5 ab
AgriCal	560/560	Early/Mid bloom	96.6 a	71.9 a	79.4 a
Lime	1120	Planting	95.9 a	76.4 a	70.3 ab
Lime/AgriCal	1120/560	Planting/Early bloom	97.9 a	67.8 a	75.3 ab
Hi-Cal	374	Early bloom			66.9 b
AgriCal/Hi-Cal	1120/37	Early/Mid bloom			69.8 b

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# IV. Soil Extraction Methods for Calcium for Peanut Production in the Coastal Plain

### Abstract

Sufficient Ca in the upper 7 to 10 cm of soil (e.g., pegging zone) is essential for peanut production. Comparison of methods among routine soil tests on pegging zone samples and their ability to supply Ca to peanut is needed. The objective of this study is to evaluate relationships and predictability of four major Ca tests including Mehlich 1 (M1), Mehlich 3 (M3), 1 N neutral ammonium acetate (AA), and 0.01 M sodium nitrate (SN) for peanut production in Coastal Plain soils. Results showed that soil test Ca extracted by M1 and M3 was similar and significantly greater than AA and SN. Application of gypsum 30 d before soil sampling had little effect on correlations among the four tests; however, application of lime led to weaker correlations. In lime-treated soils, slopes of regression lines for soil Ca among M1 and the other three tests were significantly higher than in untreated soils, suggesting M1 may overestimate soil available Ca in lime-treated soils. Soil K determined by M3 and AA was similar, but approximately 20 and 70% greater than M1 and SN method, respectively. Correlations were strong among soil K determined using M1, M3, and AA, but the relationship was weak between M1 and SN. Soil Mg determined by M1, M3, and AA was similar, while soil Mg determined by SN was considerably lower than other methods. All methods for soil Mg were strongly correlated. The AA test for Ca had the best relationships to peanut yield, grade, and seed Ca, followed by M1.

## Introduction

Adequate availability of Ca in the pegging zone (upper 7 to 10 cm of soil) is extremely important for peanut pod development and therefore quality peanut production. Many soil tests have been developed to evaluate available Ca in the soil for developing plants; however, none were specifically developed for peanut. Currently, recommendations of supplemental Ca for peanut production in many of the peanut producing states in the southeastern United States are based on the Mehlich 1 (M1) soil test (Mehlich, 1953) evaluated on pegging zone samples. The M1 test is used because it is a routine soil test for Ca, K, Mg, and P for all crops in this region, and is thus a convenient method for evaluation. Other tests, such as Mehlich 3 (M3) and 1 N neutral ammonium acetate (AA), are also widely used for Ca and may be used in other peanut producing regions (e.g., M3 in North Carolina). The M3 test was developed for a wider range of soils including calcareous soils (Mehlich, 1984), and AA was first developed to analyze exchangeable cations in the soil (Chapman, 1965). Correlations among the above-described methods for various soil nutrients have been intensively studied, and conversion equations have been provided for a wide range of soils (Hartzog and Adams, 1973; Mehlich, 1984; Michaelson et al., 1987; Sims et al., 1989; Beegle and Oravec, 1990; Evans and McGuire, 1990; Gascho et al., 1990; Gartley et al., 2002; Mylavarapu et al., 2002; Wang et al., 2004; Franklin and Simmons, 2005).

Some concerns have been raised about the accuracy of soil test Ca for supplemental Ca recommendations for peanut production. Studies have shown that M1 may overestimate available Ca to developing pods in lime-treated soils, due to the dissolution of undissolved lime by the highly acidic M1 solution (Smal et al., 1989; Alva et al., 1990). Research also found that peanuts cultivated on soils with higher extractable Ca by AA did not necessarily produce higher

yields than those grown on soils with lower AA-extractable Ca, indicating that AA-extractable Ca is a poor indicator of Ca availability to peanut (Alva et al., 1989). In order to improve the predictability of soil available Ca for peanut production, a diluted salt solution, 0.01 M sodium nitrate (SN), was proposed by Smal et al. (1989) in Georgia. Their results indicated that peanut yield, grade, and pod rot were better correlated with Ca using the SN solution than with M1 (Smal et al., 1989) using the boundary line method by Webb (1972). However, these results have not been widely verified by other researchers, and this soil Ca test has not been widely adopted by public or private soil testing programs in major peanut growing regions.

Soil Ca is not the only criteria that should be used to provide accurate Ca fertility recommendations. The Ca/K ratio also should be considered in order to determine whether a soil is limited by Ca. When the Ca/K ratio is < 3:1, the capability of peanut to absorb Ca may be limited (Alva et al., 1989). This is due to competition between Ca and K for plant uptake. High concentrations of K may also limit Ca diffusion to the pod. Sullivan et al. (1974) found that application of K fertilizers reduced yield, percentage of SMK, and proportion of extra-large kernels and tended to increase the incidence of dark plumule; however, negative effects of K application were not noted when Ca fertilizer was also applied. Conversely, over-application of Ca may limit availability of K and Mg to the peanut plant and subsequently reduce yield, especially when soil K and Mg are very low.

Because of these noted interactions between Ca and K and Mg, there have been some efforts to incorporate these factors into recommendations. In Georgia, current recommendations state that supplemental Ca for peanut is needed if soil Ca in the pegging zone is <250 mg kg<sup>-1</sup> and the Ca/K ratio is <3:1 (Kissel and Sonon, 2008). The activity ratio of Ca, defined as mol Ca per mol of the sum of Ca, K, and Mg, has also been used as an effective indicator of Ca availability for

peanut production. Optimum relative yield per fruit cavity (i.e., seed weight per number of cavities) occurred with an activity ratio of 0.25 (Wolt and Adams, 1979). Although the ratio of Ca to Mg and K has been recognized to be important for peanut production, there are few studies that have evaluated the effect of Ca fertilization on soil test Mg and K for peanut production.

Due to the differences among extractant solutions, relationships among methods in context of Ca supplementation need to be evaluated.

The objective of this study is to 1) compare relationships among the common soil Ca tests, M1, M3, AA, and SN, 2) determine effects of recent field applications of gypsum and lime on soil test Ca, Mg, and K, and 3) evaluate the ability of soil Ca tests for correct evaluation of Ca availability for peanut.

### **Materials and Methods**

Soil samples were taken from Ca supplementation field trials conducted at the Wiregrass Research and Extension Center (WREC) in Headland, AL (31.36 °N, 85.32 °W), and the Gulf Coast Research and Extension Center (GCREC), in Fairhope, AL (30.55 °N, 87.87 °W), in 2012 and 2013. Soil series were Dothan sandy loam (fine-loamy, kaolinitic, thermic Plinthic Kandiudults) and Lucy loamy sand (loamy, kaolinitic, thermic Arenic Kandiudults) at WREC, and Malbis fine sandy loam (fine-loamy, siliceous, subactive, thermic Plinthic Paleudults) at GCREC. The field trials were organized as randomized complete block designs with 4 replications under irrigated and non-irrigated conditions. Soil samples were taken from the following treatments: untreated control, 1120 kg ha<sup>-1</sup> gypsum applied at early bloom, and 1120 kg ha<sup>-1</sup> lime applied at planting. Pegging zone soil samples (top 7 cm of soil) were taken at planting, mid-bloom (approximately 70 to 80 days after planting, DAP), and harvest. All

pegging zone soil samples were composite samples of 14 individual samples collected from each plot with a hand-held soil probe, dried at  $60 \, \text{C}$  for 48 h, ground using a mortar and pestle, and sieved to pass a 2-mm sieve.

Soil Ca, Mg, and K were determined using four different soil tests: M1 (Mehlich, 1953), M3 (Mehlich, 1984), AA (Chapman, 1965), and SN (Smal, et al., 1989). For M1 and AA tests, 5 g soil was shaken in a centrifuge tube with 20 mL extracting solution for 5 min. The M3 test uses a soil/solution ratio (w/v) of 1:10, instead of 1:4. For this test, 20 mL of M3 solution was added to 2 g soil and shaken for 5 min. The suspensions from M1, M3, and AA extractions were centrifuged at 1000 rcf for 10 min and the supernatant filtered through Whatman No. 40 filter paper (GE Healthcare Life Sciences, Pittsburgh, PA). For SN test, a soil solution ratio (w/v) of 1:1 was used. Briefly, 20 g soil was shaken in a centrifuge tube with 20 mL 0.01M SN solution for 30 min, centrifuged at 1400 rcf for 20 min, and filtered with Whatman No. 42 filter paper (GE Healthcare Life Sciences, Pittsburgh, PA). The filtrate from all extractant solutions was analyzed for Ca, Mg, and K by inductively coupled plasma (ICP) spectroscopy (Spectro Ciros CCD, side on plasma, SPECTRO Analytical Instruments Inc., Kleve, Germany).

Peanut yield from sampled treatment plots was determined at harvest after peanuts were dried to approximately 10% moisture. Peanut grade represents the percentage of sound mature kernels (SMK) in a 250 g sample. The SMK were then dried at 60 °C for 48 h, finely ground in a coffee grinder (Hamilton Beach Inc., Picton, Canada), and microwave digested using a Mars Xpress microwave (CEM Corp., Matthews, NC) using the modified EPA 3051A procedure (USEPA, 2007) prior to Ca determination by ICP. In detail, 1 g finely ground peanut sample was pre-digested overnight in 10 mL 30% (v/v) hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) and then microwave digested for 15 min in 10 mL concentrated nitric acid (HNO<sub>3</sub>). The final digested material was

filtered using Whatman No. 40 filter papers, and the volume was adjusted to 100 mL with deionized water before analysis by ICP. In order to combine yield data from multiple years and growing conditions, relative yields were calculated for each experimental plot. Relative yield represents the yield of the plot compared to the mean yield of the untreated control treatment for that experimental site and year.

In order to evaluate the extraction of Ca by various amendments, the recovery of Ca from AgriCal, USG 500, PCS Wetbulk, and agricultural lime in M1, M3, AA, and SN was studied. A solution to solid ratio >1000:1 was used to assure that the solubility of gypsum and lime was not limiting Ca recovery. In detail, 200 mg gypsum material was measured and shaken with 250 mL extractant solution for 2 h, while 4 mg lime material was measured and shaken with 500 mL extractant solution. The suspension was allowed to settle for 1 h and the supernatant was filtered using Whatman No. 40 filter paper. Concentration of Ca in filtrate was analyzed by ICP. Three analytical replications were performed for each amendment. Total Ca in each of the materials was performed similarly to seed Ca using the EPA 3051A procedure without the hydrogen peroxide pretreatment.

Biological processes in nature are very complex, and many statistical models (e.g., linear and non-linear regressions) have been developed to understand the variability of a biological response. However, traditional regression methods basically explain the average response of a variable against a covariate or a set of covariates and may be meaningless, unless the dependent variable can be treated as an additive combination of all explanatory variables in a biological context (Milne et al., 2006). Webb (1972) suggested that the upper or lower boundary of a plot may be used for better explanation of biological response. Biologists have found that boundary line model is persuasive and of practical interest in various conditions. This conceptual model

has been applied to evaluate the cause-effect relationships in biological data in crop growing environments (Casanova et al., 1999; Schmidt et al., 2000; Shatar and McBratney, 2004; Lewandowski and Schmidt, 2006). In the current study, the boundary line method developed by Webb (1972) was used to evaluate correlations between soil-test Ca and relative yield, grade, and seed Ca concentration. In brief, outliers were first detected and removed. Data points with the largest 5% of Mahalanobis distances were removed (Shatar and McBratney, 2004). Then, each soil Ca value was divided into 20 units containing equal number of data points. Maximum relative yield, grade, seed Ca concentration, and corresponding soil Ca value within each unit were identified and combined to form a data subset. The boundary line was fitted using the data subset in Sigma Plot 12.5 software (Systat Software, San Jose, CA). Boundary line equations and adjusted coefficient of determination (r²) were reported.

# **Results and discussion**

# Evaluation of soil test Ca methods

Soils from plots receiving no Ca supplements,  $1120 \text{ kg ha}^{-1}$  gypsum at early bloom, and  $1120 \text{ kg ha}^{-1}$  lime at planting were evaluated for soil test Ca, K, and Mg using the M1, M3, AA, and SN methods. The soil test method significantly affected soil test Ca (P < 0.001). However, no significant Ca treatment effect (P = 0.282) or Ca treatment  $\times$  soil test method interaction (P = 0.604) was observed; therefore, data were combined for all Ca treatments to evaluate effect of soil test method on soil test Ca (Figure 5). Soil test Ca using the M1 and M3 methods was similar and significantly greater than the AA and SN methods. The AA test extracted <70% and the SN test <9% of the Ca that was extracted by M1 and M3.

Even though there was no Ca treatment × soil test method interaction, the relationships among Ca extracted by M1, M3, AA, and SN may differ; therefore, it was of interest to evaluate the regression among different methods in order to establish conversion equations among the methods. Soils taken at mid-bloom from the following treatments were used: untreated control, 1120 kg ha<sup>-1</sup> gypsum applied at mid bloom, and 1120 kg ha<sup>-1</sup> lime applied at planting. The correlations between M1 and M3 ( $r^2 > 0.9$ ), M1 and AA ( $r^2 > 0.9$ ), M1 and SN ( $r^2 > 0.8$ ) were strong in untreated and gypsum-treated soils (Figure 6); however, application of lime notably weakened the correlations, especially between M1 and SN ( $r^2 = 0.51$ ; Figure 6J). These results indicate that conversion of data from one method to another is less accurate in lime-treated soils compared to untreated or gypsum-treated soils. Results also found that slopes of regression lines for soil Ca in untreated and gypsum-treated soils between M1 and M3 (P = 0.781), M1 and AA (P = 0.348), and M1 and SN (P = 0.808) were not significantly different, suggesting that applied gypsum had little effect on relationship among soil Ca tests. Since the solubility of gypsum (2.6 g L<sup>-1</sup>) is relatively high, gypsum dissolution is likely to have occurred in the field or during extraction with the soil test. This is especially true in this case because a finely powdered form of gypsum was used.

In order to more thoroughly evaluate the effect of gypsum contribution to soil test Ca, the tests were evaluated directly on three different gypsum sources. Recovery of Ca from AgriCal, which is the gypsum used in this study, did not differ using M1, AA, and SN methods (Table 14). Alva et al. (1989) compared recovery rate of seven gypsum materials with various particle sizes and also found that the recovery of Ca in M1 and SN for finely powdered gypsum was not significantly different. Therefore, it is likely that if any undissolved gypsum particles were present in the soil sample, the M1, AA, and SN methods were able to completely dissolve them,

and the relationships among the tests would not be significantly affected. Recovery of Ca using the M3 method was significantly lower (56%) than with the other tests. This is not consistent with comparison of M1 and M3 methods, which were approximately the same. In order to better understand this contradictory issue, the chemical speciation program MINTEQA2 (USEPA, 2006) was used to calculate the composition of equilibrated M1, M3, AA, and SN solution after shaking with gypsum. Results showed that fluoride (F<sup>-</sup>), which is dissociated from ammonium fluoride (NH<sub>4</sub>F) in M3 solution could react with Ca from gypsum and form calcium fluoride (CaF<sub>2</sub>) precipitant, which would be removed through filtration. Therefore, the M3 test recovered less Ca from gypsum than the other tests. In the current study, the mid-bloom soil samples were taken 30 days after gypsum was applied. A majority of gypsum may have been dissolved with sufficient rainfall and absorbed by peanut or leached out of the pegging zone, thus inclusion of undissolved gypsum particles is less likely in this study. Therefore, correlations for soil test Ca among different tests were similar in untreated and gypsum-treated soils.

The slopes of regression lines for soil test Ca in lime-treated soils between M1 and M3 (P < 0.001), M1 and AA (P = 0.001), and M1 and SN (P < 0.001) were significantly higher than in untreated and gypsum-lime soils (Figure 6). These results suggest that relationships among methods is different when lime has been applied within the last 6 months. This indicates that M1 extracts more Ca than M3, AA, and SN in lime-treated soils than in untreated or gypsum-treated soils. Alva et al. (1990) reached the same conclusion using a greenhouse incubation study, which had relatively uniform initial soil Ca in each pot since soils were thoroughly mixed before the study. In current field study, however, initial soil Ca varied among locations and within each location. For example, initial soil Ca at WREC was significantly lower than at GCREC, and untreated control plots had significantly higher background Ca than gypsum-treated plots in 2012

at WREC. Varied soil Ca levels may affect correlations; therefore, samples taken at planting and at mid-bloom from gypsum- and lime-treated plots were used to further verify these results. Soils taken at planting before gypsum and lime application were defined as pre-gypsum and prelime, while those taken at mid bloom after gypsum and lime application were defined as postgypsum and post-lime. Using this method of comparison, variation among plots can be reduced. Similar results were noted. Slopes of regression lines between M1 and M3 (P = 0.808), M1 and AA (P = 0.659), and M1 and SN (P = 0.884) in pre- and post-gypsum soils did not differ significantly (Figure 7), whereas slopes of regression lines between M1 and M3 (P = 0.013), M1 and AA (P = 0.019), and M1 and SN (P < 0.001) in post-lime soils were significantly higher than in pre-lime soils (Figure 8). These results can be attributed to inclusion of undissolved lime in the soils, because lime has a lower solubility (0.01 g L<sup>-1</sup>) than gypsum (2.6 g L<sup>-1</sup>). Since M1 has a pH between 1 and 2, recovery of Ca from two types of agricultural lime using the M1 method was 25, 50, and 200% greater than with M3, AA, and SN (Table 14). In addition, strongly retained Ca on the exchange complex may be extractable with M1, but it is not extractable in dilute salt solutions such as SN (Alva et al., 1990). Therefore, M1 could overestimate available soil Ca in lime-treated soils, compared to M1, AA, and SN. The likelihood and magnitude of overestimation may depend on the time of application and particle size of the liming material. If lime is applied well in advance of the date of sampling (e.g., 6 mo before the sampling date), undissolved lime in soil samples is unlikely (Smal et al., 1989; Alva et al., 1990). Particle size of lime may also affect dissolution and thus the chance of undissolved lime in soils, because fine particles are more easily dissolved than coarse particles. More than 50% of particles in the lime used in the current study was < 1 mm. A large proportion of lime may have been dissolved with

rainfall, especially in 2013, which was a particularly wet year. Therefore, unrealistically high M1-Ca in lime-amended soils was not observed in this study, but could be a factor in drier years.

# Soil Test Mg and K

Soil test method (P < 0.01) and Ca treatments (P < 0.01) significantly affected soil test K and Mg. However, no significant Ca treatment  $\times$  method interaction (P > 0.5) was observed. Data were combined for all soil test methods to evaluate effect of Ca supplementation on soil extractable K and Mg. The effect of soil test method on extraction of K showed that M3 and AA were very similar and significantly higher than M1 and SN. The M1 test for K was 80% of M3, and the SN test was 30% of M3 (Figure 9A). For Mg, there was no significant difference among M1, M3, and AA tests; however, Mg with SN was only 14 to 17% of that with the other three methods (Figure 9B).

Addition of gypsum and lime significantly decreased soil Mg and K content compared to the untreated control (Figure 10). Previous studies have also reported that high Ca fertilization may reduce availability of K and Mg in the pegging zone, possibly due to competition among Ca, K, and Mg for plant absorption (Alva and Gascho, 1991; Hallock and Allison, 1980b).

Due to lack of treatment  $\times$  method interaction, data were combined for all Ca treatments to evaluate regression equations for soil test K and Mg extractable by M1, M3, AA, and SN. There were strong correlations for K extracted by M1, M3, and AA ( $r^2 > 0.7$ ); however, M1 and SN were poorly correlated ( $r^2 = 0.37$ , Figure 11C). Strong correlations between M1 and SN for K have been reported (Alva et al., 1989; Alva et al., 1990). However, these studies used a soil/solution ratio of 1:4 for SN extraction rather than the 1:1 ratio reported in the original method (Smal et al., 1989) and used in the current study. Different soil solution ratios can affect

the extraction process and result in different correlations. There were strong correlations ( $r^2 \ge 0.9$ ) among soil Mg extracted by M1, M3, AA, and SN (Figure 12).

# Correlations between soil Ca and relative yield, grade, and seed Ca

Relationships between soil test Ca and relative yield of peanut were weak. For relative yield of peanut, coefficients of determination (r<sup>2</sup>) were 0.49, 0.46, 0.70, and 0.28 for M1, M3, AA, and SN, respectively (Figure 13). Peanut grade also did not have a strong relationship with soil test Ca. Coefficients of determination (r<sup>2</sup>) were 0.55, 0.39, 0.59, and 0.44 for M1, M3, AA, and SN, respectively (Figure 14). These results indicate that the AA test was the best predictor of yield and grade and that M1 was the next best predictor. The SN test was not a particularly good indicator of either yield or grade. Alva et al. (1989) reached the same conclusion; however, Smal et al. (1989) reported a better relationship between SN and yield and grade than M1.

Several factors could account for the poor relationships between soil tests and peanut yield and grade as well as the variable results from various studies. In the current study, the background Ca level was similar or greater than the established critical value (150 mg kg<sup>-1</sup>) for peanut production in Alabama. Therefore, Ca may not be a limiting factor for peanut production, and a yield or grade response to increased soil Ca may not occur. Also, there could be an effect of peanut variety. Each of these evaluations was performed with different peanut cultivars that may have different Ca uptake patterns or requirements.

Strong correlations ( $r^2 > 0.6$ ) were found between seed Ca concentration and soil Ca using the different test methods (Figure 15). The AA test had the best correlation with seed Ca ( $r^2 = 0.81$ , Figure 15C). Seed Ca concentration increased with increasing soil Ca extracted by M1, M3, and AA from 150 to 300 mg kg<sup>-1</sup>, suggesting that seed Ca concentration can further increase

to a level above that which is required for optimum yield and grade. This positive relationship between soil Ca and seed Ca has been previously reported. Adams et al. (1993) reported correlation coefficients between soil Ca and seed Ca were 0.49, 0.51, 0.52, and 0.54 for four different runner-type peanuts, including GK7, Florunner, Southern Runner, and Sunrunner, respectively. A lower correlation coefficient of 0.40 was observed by Adams and Hartzog (1991), but authors argued that this correlation may be weakened by various moisture regimes of the peanuts used in the study. Since higher seed Ca corresponds to improved germination, growers producing peanuts for seed are advised by Auburn University (Mitchell, 1994) in Alabama and University of Georgia (Kissel and Sonon, 2008) in Georgia to apply gypsum at bloom even if soil Ca levels are above the critical value for optimum peanut yield.

## **Conclusion**

The M1 and M3 methods extract similar amounts of soil Ca, and both tests extract significantly higher amounts of soil Ca than AA and SN. Addition of gypsum 30 d before soil sampling had minimal effect on correlations among soil test Ca. In addition, strong correlations for soil Ca exist with M1, M3, AA, and AA in gypsum-treated soils; therefore, any method can be used if gypsum was used as Ca supplementation.

The M1 test might overestimate soil Ca in lime-treated soils, if undissolved lime is present in the soil samples. This is because recovery of lime with M1 is significantly higher than with M3, AA, and SN. Therefore, soil Ca extracted by M1 is accurate only when no lime was applied before soil sampling or when lime was applied well in advance of soil sampling date (e.g., 6 mo before soil sampling).

All the four tests can be used for soil K except SN, because the SN test for soil K was poorly correlated with M1 test. Strong correlations exist among soil test Mg with M1, M3, AA, and SN; therefore, either test can be used and converted interchangeably.

The soil Ca extracted by AA showed the best correlation with peanut yield, grade, and seed Ca concentration among the four evaluated tests. The M1 test is the second best, and the SN test was the worst. In practice, M1 test may be used for routine soil test in order to improve the efficiency of soil testing, since M1 extracts nutrients other than Ca (i.e., P, Mg, and K) at the same time. The soil test Ca using M1 can be converted to AA-extractable Ca, because soil test Ca with M1 and AA is strongly correlated. This way, the accuracy for peanut supplemental Ca recommendation can be improved as well.

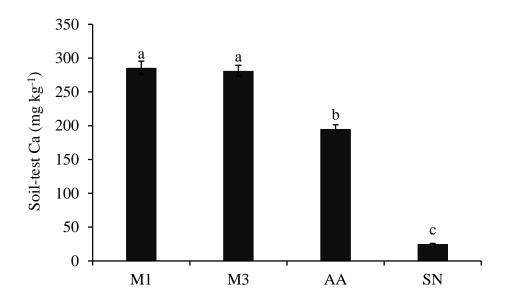


Figure 5. Soil-test Ca extractable by Mehlich 1 (M1), Mehlich 3 (M3), 1 N neutral ammonium acetate (AA), 0.01 M sodium nitrate (SN) methods. Data were combined for all treatments due to lack of treatment  $\times$  method interactions. Different letters across bars indicate not significantly difference at  $\alpha$ =0.05.

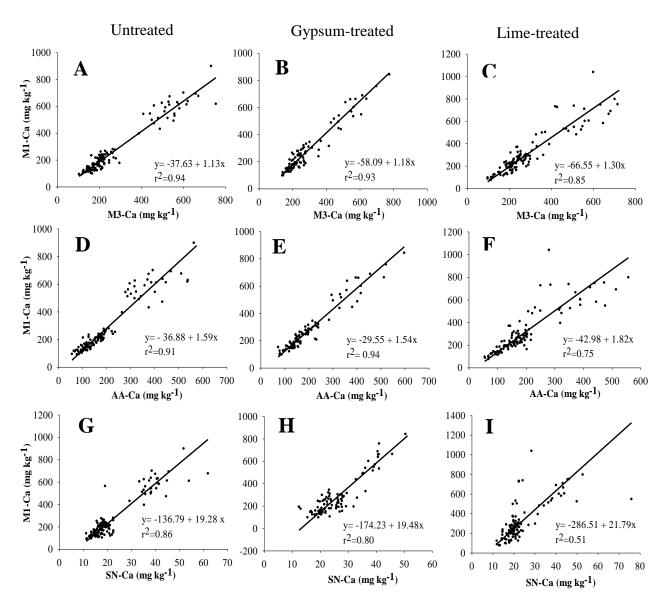


Figure 6. Relationships between soil Ca extracted by Mehlich 1 (M1), Mehlich 3 (M3), 1 N neutral ammonium acetate (AA), and 0.01 M sodium nitrate (SN) methods in untreated, gypsumtreated, and lime-treated soils at Wiregrass Research and Extension Center (WREC) and Gulf Coast Research and Extension Center (GCREC) in 2012 and 2013.

Table 14. Recovery of Ca from three gypsum sources, AgriCal, USG 500, PCS Wetbulk, and two types of agricultural lime using the Mehlich 1 (M1), Mehlich 3 (M3), 1 N neutral ammonium acetate (AA), and 0.01 M sodium nitrate (SN) soil test methods. Calcium recovered in each extractant solution was calculated as a percentage of total Ca in the gypsum and lime materials. Means within each column with the same letters indicate not significantly different at  $\alpha$ =0.05.

Soil test	AgriCal	USG 500	PCS Wetbulk	Calcitic Lime	Dolomitic Lime
			Recovery of C		
			%		
M1	94.5 a	94.1 a	97.9 a	99.7 a	99.9 a
M3	55.8 b	56.9 b	77.8 b	88.4 b	83.2 b
AA	87.5 a	93.6 a	93.4 a	73.6 b	80.8 b
SN	92.2 a	86.1 a	94.8 a	35.1 c	46.9 c

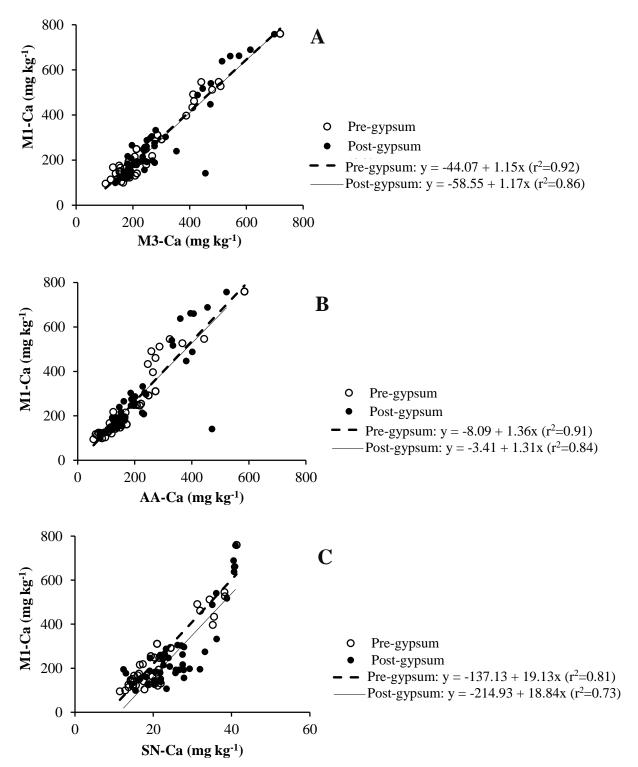


Figure 7. Relationships between soil Ca extracted by Mehlich 1 (M1), Mehlich 3 (M3), 1 N neutral ammonium acetate (AA), and 0.01 M sodium nitrate (SN) methods in untreated and gypsum-treated soils. Pre- and post-gypsum data points represent soil samples taken before and after gypsum application.

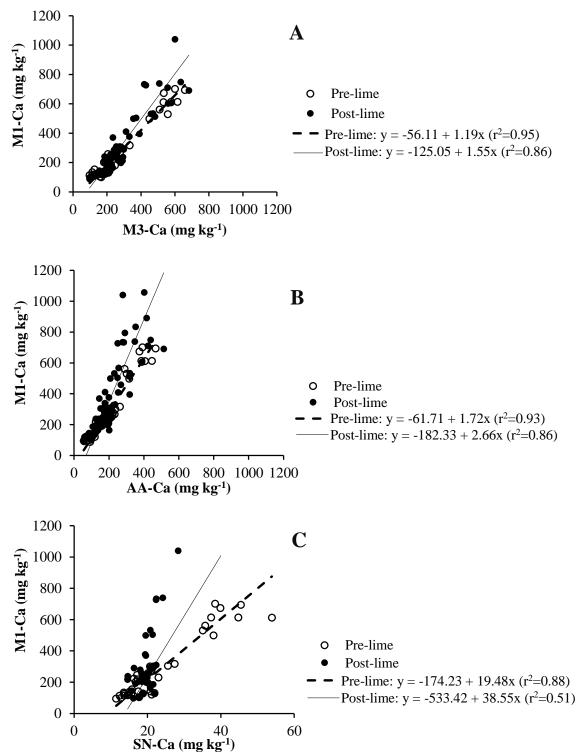


Figure 8. Relationships between soil Ca extracted by Mehlich 1 (M1), Mehlich 3 (M3), 1 N neutral ammonium acetate (AA), and 0.01 M sodium nitrate (SN) methods in untreated and limetreated soils. Pre- and post-lime data points represent soil samples taken before and after lime application.

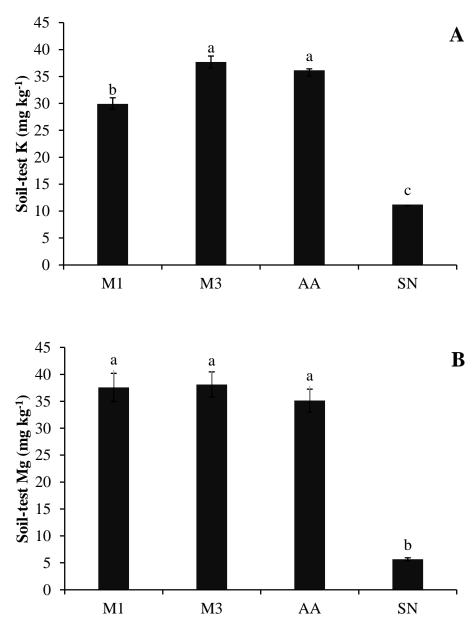


Figure 9. Soil test K and Mg extractable by Mehlich 1 (M1), Mehlich 3 (M3), 1 N neutral ammonium acetate (AA), 0.01 M sodium nitrate (SN) methods. Data were combined for all treatments due to lack of treatment  $\times$  method interactions. Different letters across bars indicate not significantly difference at  $\alpha$ =0.05.

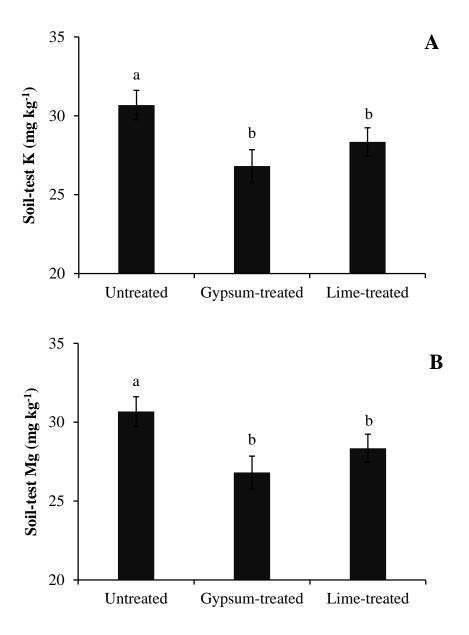


Figure 10. Soil test K in soils amended with no Ca, 1120 kg ha<sup>-1</sup> gypsum at early bloom, and 1120 kg ha<sup>-1</sup> lime at planting. Data were combined for all soil test methods due to lack of treatment  $\times$  method interactions. Different letters across bars indicate not significantly difference at  $\alpha$ =0.05.

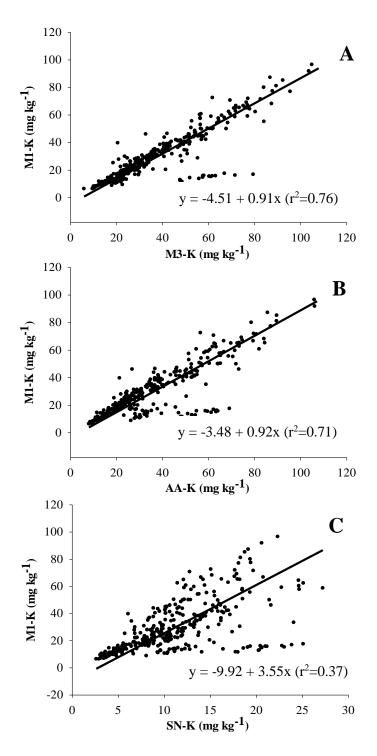


Figure 11. Relationships between soil K extracted by Mehlich 1 (M1), Mehlich 3 (M3), 1 N neutral ammonium acetate (AA), and 0.01 M sodium nitrate (SN) methods at Wiregrass Research and Extension Center (WREC) and Gulf Coast Research and Extension Center (GCREC) in 2012 and 2013. Data were combined for all treatments due to lack of treatment × method interaction.

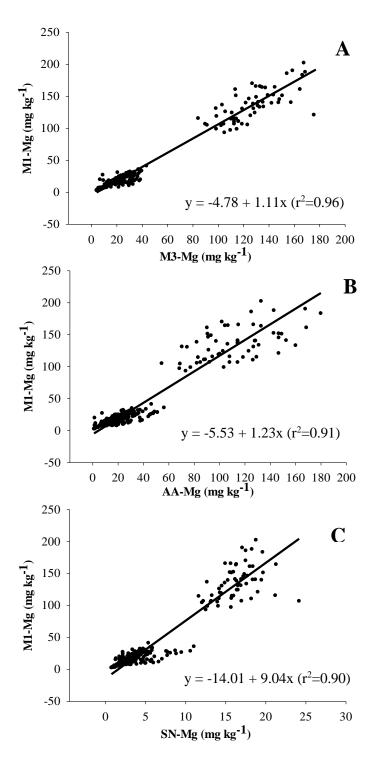


Figure 12. Relationships between soil Mg extracted by Mehlich 1 (M1), Mehlich 3 (M3), 1 N neutral ammonium acetate (AA), and 0.01 M sodium nitrate (SN) methods at Wiregrass Research and Extension Center (WREC) and Gulf Coast Research and Extension Center (GCREC) in 2012 and 2013. Data were combined for all treatments due to lack of treatment × method interaction.

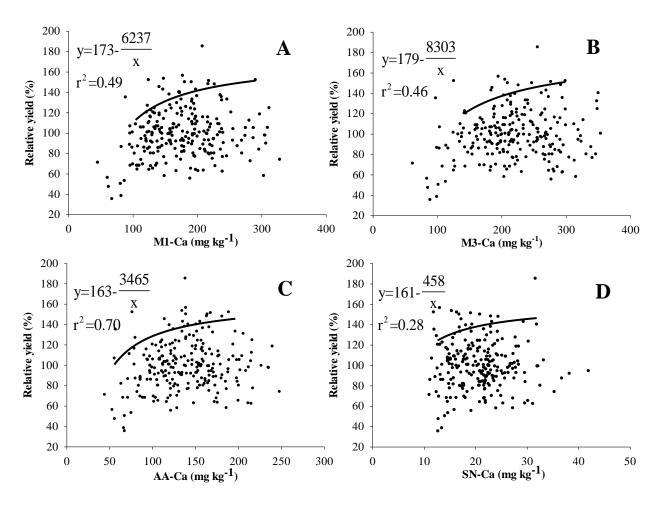


Figure 13. Correlations among peanut relative yield and soil extractable Ca by Mehlich 1 (M1), Mehlich 3 (M3), 1N neutral ammonium acetate (AA), and 0.01 M sodium nitrate (SN) generated by boundary line method by Webb (1972). Data for all Ca treatments and all cultivars under irrigated and non-irrigated trials at WREC in 2012 and 2013 were combined.

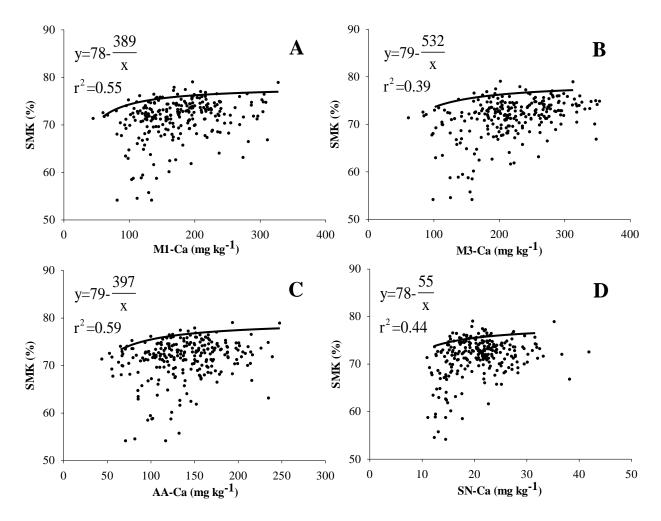


Figure 14. Correlations among peanut grade represented as percentage of sound mature kernels (SMK) and soil extractable Ca by Mehlich 1 (M1), Mehlich 3 (M3), 1N neutral ammonium acetate (AA), and 0.01 M sodium nitrate (SN) generated by boundary line method by Webb (1972). Data for all Ca treatments and all cultivars under irrigated and non-irrigated trials at WREC in 2012 and 2013 were combined.

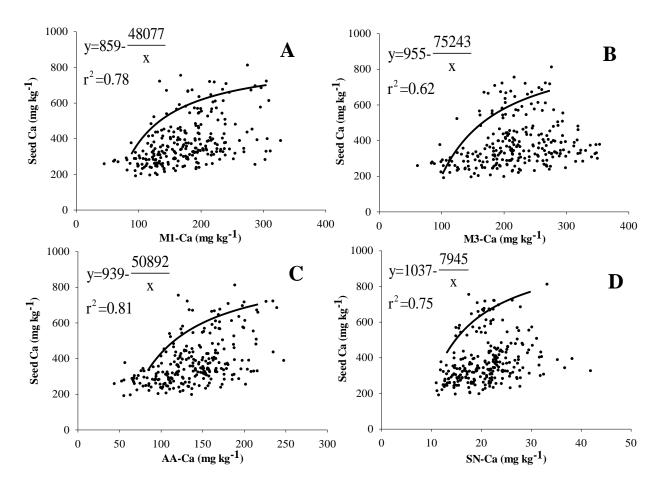


Figure 15. Correlations among peanut seed Ca concentration and soil extractable Ca by Mehlich 1 (M1), Mehlich 3 (M3), 1N neutral ammonium acetate (AA), and 0.01 M sodium nitrate (SN) generated by boundary line method by Webb (1972). Data for all Ca treatments and all cultivars under irrigated and non-irrigated trials at WREC in 2012 and 2013 were combined.

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### V. Conclusion

Overall, it is important to consider providing adequate Ca throughout the pod development period. Seed Ca concentration is relatively consistent during the 4 wk period prior to harvest, and can be used to predict seed quality, which allows for possible management practice to increase total seed Ca and germination before harvest.

Application of slow release Ca supplements such as lime is very effective when rainfall is plentiful; however, dry years may benefit from gypsum applications. Any type of gypsum evaluated in this study can be used by peanut producers, but USG 500 is recommended while PCS Wetbulk is less preferred. Liquid Ca fertilizers (e.g., Hi-Cal) are not recommended for peanut producers because of its low efficacy due to the low total Ca input and high leaching potential. Split applications of gypsum at early/mid bloom show promise as a mechanism to improve seed quality parameters such as seed Ca and germination.

Methods for soil Ca, K, and Mg such as M1, M3 and AA are well correlated, but M1 could overestimate soil available Ca in lime-amended soils; therefore, M1 should not be used within 6 mo of lime application. The AA-extractable Ca had the best correlations with peanut yield, grade, and seed Ca concentrations, and thus can be used to improve predictability of supplemental Ca for peanut production. The SN test was poorly correlated to other methods and to peanut production evaluation characteristics.