

UTILIZING DDGS AND CRUDE GLYCEROL IN ANIMAL DIETS: FEED
MANUFACTURING CONSIDERATIONS

by

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Abstract

Three experiments were conducted to evaluate value added uses for dried distillers grains with solubles (DDGS) and crude glycerol in swine and poultry diets. In Exp.1, crude glycerol from multiple biodiesel production facilities was evaluated for storage and variability characteristics. Part one reviewed the storage capabilities of crude glycerol at room temperature and excessive heat conditions on ferrous and stainless steel metal. There was no notable corrosion during the two-month experiment for either metal type under each condition. There was a slight discoloration observed on the ferrous metal stored under excessive heat conditions, but no pitting or sign of corrosion was noted. No changes were observed in the stainless steel under either environment condition. Part two evaluated the variability of crude glycerol from multiple biodiesel production facilities. Representative samples of the parent feedstock and resultant glycerol was collected and analyzed. There was considerable variation between samples, particularly when comparing glycerol from the different feedstocks (vegetable vs. animal). Exp. 2 and 3 took place in the Feed Processing Research Center in the Department of Grain Science at Kansas State University. Pellet quality and electrical energy consumption was evaluated by reviewing production rate, conditioning and hot pellet temperatures, motor load, and pellet durability index (PDI) as testing parameters. In Exp. 2, diets containing varying levels of crude glycerol were evaluated on pellet quality and pellet mill performance in a pilot mill and in a commercial facility. For part one, a corn-soy based swine grower diet was formulated to contain 0, 3, 6, and 9% crude glycerol. Each diet was steam conditioned to 150, 170 and 190 °F in an atmospheric conditioner and pelleted. An interaction existed between glycerol and conditioning temperature. For all diets containing glycerol, roll skid occurred and the pellet mill plugged as conditioning temperature approached 190°F. Pellet quality increased linearly ($P < 0.01$) with increasing levels of glycerol. Part two took place at Don's Farm Supply in Newell, IA. A corn-soy based turkey grower diet was formulated to contain 3% glycerol and pelleted. Results from part two were consistent with part one, in that there was an interaction between conditioning temperature and the addition of glycerol. Exp. 3 evaluated DDGS on pellet quality and electrical consumption. A poultry diet was formulated to contain 0, 15, or 30% DDGS and steam conditioned to 140, 160, and 180°F. As conditioning temperature increased, pellet quality in diets containing DDGS significantly improved ($P > .001$). Electrical consumption in diets

containing DDGS compared to the control showed no significance in reduction in energy usage ($P > .001$). In conclusion, the production of pelleted diets containing crude glycerol is a feasible option; however, conditioning temperatures should be kept minimal and storage and variation of the material should be considered. In addition, improved pellet quality in pelleted diets containing DDGS is in part by conditioning at higher temperatures, respectively. The data suggests that the addition of crude glycerol and DDGS in animal diets can serve as a beneficial feed additive.

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Introduction

Replacing petroleum based fuels with ethanol or biodiesel is certainly not a new concept. About a century ago, biofuels were used to power the first automobiles; Henry Ford's Model T design was built to run on pure ethanol, and a few years later, Rudolph Diesel developed an engine to run on peanut oil (Grillo, 2008). Ethanol and biodiesel continued to fuel early vehicles and serve non-transportation wartime uses for several years until large petroleum deposits were discovered and gasoline and diesel were kept cheap for several decades. In 1973, due to the oil embargo, the Organization of Petroleum Exporting Countries (OPEC) increased the price of oil by 70% overnight, shocking the U.S. and forcing the population to go into conservation mode (Yergin, 2008). The recognition of the reliance on foreign oil surfaced, and alternative energy resources such as ethanol and biodiesel were reintroduced (Grillo, 2008). Moreover, ultimately the volatility of the global oil markets in part contributed to the conflicts in Iraq and Afghanistan and resulted in the developments of U.S. energy policies and the rapid growth of the biofuels industry (Teslik, 2008).

In 1992, the Energy Policy Act (EPAct) set goals, established mandates and amended utility laws in efforts to increase clean energy and improve overall energy efficiency in the United States (Kenney et al. 2008). The EPAct took responsibility for implementing specific standards requiring the development and use of alternative resources that could be utilized as renewable fuels. In 2005, the Renewable Fuel Standard program was created under the EPAct, which increased the amount of renewable fuel required to be blended into gasoline to 7.5 billion gallons by 2012. After a number of amended rules and regulations to the Renewable Fuel Standard, in 2007 it was developed into the Energy Independence and Security Act (EISA), which in conjunction with the Renewable Fuel Standard, appointed the EPA responsible for

requiring a minimum volume of renewable fuel sold in the United States each year. The new projected amount would increase the volume of renewable fuel required to be blended into transportation fuel from 9 billion gallons in 2008 to 36 billion gallons by 2022 (Renewable Fuels Association).

In 2004, the government issued the Volumetric Ethanol Excise Tax Credit (VEETC), which included a biodiesel tax credit that provided an incentive per gallon of biodiesel produced to make biodiesel more cost competitive with diesel fuel. Through this bill, blenders are allowed a credit of \$1.00 per gallon of biodiesel made from oil crops and animal fats and a \$0.50 per gallon credit for biodiesel made from recycled fats and oils. The Energy Policy Act of 2005 (PL 109-58, H.R. 6) extended this tax credit through 2008 (Yacobucci, 2007). These policies and tax incentives for ethanol and biodiesel production have contributed to the urgency in the industry to construct, and bring online, new ethanol and biodiesel facilities. Unfortunately, very little attention was given to the quantity or quality of co-products produced during the two processes, resulting in a huge waste stream.

Distillers dried grains with solubles (DDGS) and crude glycerol are the two major co-products resulting from the production of ethanol and biodiesel, respectively. Both can be produced from a variety of feedstock, and the processes required to produce ethanol and biodiesel will be provided in detail in subsequent chapters. As capacities in the biofuels industry grew, markets for these co-products quickly became saturated, and the economic value of these co-products to the facilities was marginal at best. Consequently, the rapid growth placed enormous pressure on the demand for these commodities, and it created substantial volatility in the commodity and livestock market.

The feed and livestock industry viewed the use of these inexpensive co-products as an opportunity to cheapen diet costs by incorporating them into animal rations. Additionally, as the biofuels industry began to mature and the margins for ethanol and biodiesel continued to tighten, more emphasis has been placed on adding value to the co-products by improving quality and consistency (Batal, 2009). Numerous experiments have been conducted evaluating the nutritional value of DDGS and glycerol in both monogastric and ruminant animal diets and both were shown to be effective as feed additives. However, very little work was done to evaluate including DDGS and glycerol in animal diets to improve feed quality or efficiency of feed production. Therefore, this thesis aims to determine that crude glycerol or DDGS is an economically feasible ingredient in animal feed diets that improves manufacturing characteristics and pellet quality.

Literature Review

Background and Growth

Glycerol

Biodiesel can be produced through a chemical process called transesterification, which chemically alters organically derived oils (e.g. soybean, animal fat, etc.) forming biodiesel fuel and resulting in approximately 10% glycerol by weight. For example, one hundred pounds of fat or oil (such as soybean oil) are reacted with 10 pounds of a short chain alcohol in the presence of a catalyst to produce 10 pounds of glycerin and 100 pounds of biodiesel. Biodiesel is currently considered the most appealing alternative fuel option due to its biodegradable and nontoxic composition as well as its ability to completely displace its petroleum counterpart (Johnson et al. 2007). According to the U.S. Department of Energy (DOE), biodiesel burns fewer emissions and can be used as a blend in ranges of B5-100 in diesel engine models 1992 or newer and actually improves the functionality of the diesel engine. Petroleum based fuels can leave deposits inside fuel lines, storage tanks, and fuel delivery systems over time while biodiesel serves as a lubricant, has better solvent properties, and has shown to dissolve sediments, resulting in a cleaner system. Most recently, the VEETC tax incentive is scheduled to expire on December 31, 2010. Unfortunately, with the significant delay in renewal of the policy this year, approximately only 30% of biodiesel facilities nationwide have been able to remain in production. However, the RFA is diligently working to secure passage of the extension of the .45 cent VEETC tax credit through 2015 (Renewable Fuels Association).

Nonetheless, for 2011 the EPA has set the required production of domestic biodiesel use at 800 million gallons resulting in nearly 80 million gallons of glycerol, the principal by-product of biodiesel production. Glycerol is a very versatile, nontoxic, viscous liquid used in many commercial and industrial applications. With the exponential increase in production of biodiesel throughout the past several years, the current market for glycerol has become saturated, and a large surplus of glycerin has resulted (Pagliaro et al. 2008). Therefore, utilizing the cheap, available by-product has been of interest in a number of industries, especially the animal feed industry due to its ability to feed crude glycerol as an inexpensive energy source. Thus, utilizing crude glycerol as an animal feed ingredient positions it in a new and very appealing value added market segment.

DDGS

In addition to biodiesel, ethanol is another renewable fuel on the market contributing to the Renewable Fuel Standard. During ethanol production, a feedstock, typically corn or sorghum goes through a number of processing steps resulting in end by-products of carbon dioxide and distillers grains, which are generally in the form of Dried Distillers Grains with Solubles (DDGS). Traditionally, researchers assume that for every 2.7 gallons of ethanol, 18 pounds of distillers grains are produced (Feed International, 2007). According to the Renewable Fuels Association, in 2009, ethanol biorefineries converted 3.8 billion bushels of corn into an estimated 10.6 billion gallons of ethanol and 30.5 million metric tons of high-value livestock feed, DDGS, and corn gluten feed and meal. These numbers are only projected to increase with the prodigious amount of ongoing research, innovation, and new technology.

Due to the increase in ethanol production, the amount of DDGS available has increased, and the cost has greatly reduced, giving the ingredient more potential for a variety of

applications. DDGS have been on the market in the feed industry and fed to animals for decades and serve as a valuable source of energy and protein and can be formulated for monogastric and ruminant diets, respectively. DDGS are most commonly included in animal diets as a portion of their energy source, replacing corn or soybean meal. However, inclusion levels of DDGS in animal diets tend to be restricted due to difficult processing and handling characteristics that hinder the ability to produce feed containing DDGS cost effectively.

Incorporating ingredients such as crude glycerol and DDGS in animal feed diets are very appealing to the feed industry mainly due to the ability to utilize it as a cheap energy source and the availability of the ingredient; however, it is important to consider the processing characteristics of the material. For instance, research has shown that pelleting significantly improves handling characteristics; however, multiple factors need to be evaluated that are specific to each ingredient in order to optimize production efficiencies.

Nutrition and Feeding

Glycerol

Glycerol metabolism occurs mostly in the liver and kidneys (Lin, 1977). Once glycerol is absorbed, it can be either converted into glucose via gluconeogenesis or oxidized for energy production via glycolysis and the citric acid cycle (Robergs et al. 1998). Since glycerol gluconeogenesis is generally limited by the availability of glycerol, crude glycerin has the potential of being a valuable dietary energy source for monogastrics (Kerr et al. 2007). Following digestion, intestinal absorption of glycerol has been shown to range from 70-90% in rats (Lin, 1977) and more than 97% in laying hens (Bartelt et al. 2002). Lammers et al. (2008) reported that crude glycerol, 86.95% pure, has a metabolizable energy content of $1,456 \pm 4.54$ kcal/lb, which is 94% of the metabolizable energy content of corn.

As indicated by the Association of American Feed Control Officials (AAFCO), crude glycerol has been classified as a substance that is “Generally Recognized as Safe in Animal Feeds (GRAS)” when used in agreement with good manufacturing or feeding practices. Previous studies have indicated crude glycerol to be a valuable ingredient, as a corn replacement in levels up to 10% in monogastric and ruminant diets. Also, the nutritional value of glycerol has been evaluated in numerous studies throughout the past several years focusing mainly on swine, (Kijora et al., 2006; Lammers et al., 2009), poultry (Swiatkiewics et al. 2008; Lammers et al., 2007, 2008; Cerrate et al., 2006) and most recently, ruminants (Schroder et al. 1999; DeFrain et al., 2004; Donkin et al., 2009).

Kijora et al. (2006) formulated diets containing up to 10% crude glycerol and showed it serves as a positive addition to a swine diet when fattening pigs. Feed intake improved an average of 106% in the growing period and 104% in the finishing period, which was consistent among all glycerol fed groups. There was a strong correlation between glycerol intake and live weight gain with up to 10% glycerol in the diet, regardless of purity. Lammers et al. (2009) fed isocaloric diets containing 0 to 10% crude glycerin, which were fed to growing-finishing pigs. They found that feeding glycerol caused no effect on carcass composition, growth performance, meat quality, or lesion scores in the eye, liver or kidney tissue, thus determining that crude glycerin is a viable source of dietary energy that is well utilized by pigs. Additionally, Kerr et al. (2007) showed that pigs would readily eat diets with crude glycerol because such diets had less feed dust, and corn-soybean meal diets including 10% crude glycerol flowed normally in feeders during research trials. Since the energy content of crude glycerol was also determined to be comparable to the energy present in corn, swine can readily utilize it without increasing the amount of nitrogen and phosphorus fed to or excreted.

Adding glycerol up to a level of 5% has shown no adverse effects on growth or carcass yield in poultry (Simon et al., 1996; Cerrate et al., 2006). Broiler chickens fed 2.5% and 5% crude glycerol experienced similar performance and improved breast meat yield in comparison with birds fed the control diet; however, the higher levels of glycerin (>10%) negatively affected breast meat weight, dressing percentage, body weight gain, conversion and feed consumption. Some would assume these reactions were due to the lower pellet quality and flow rate of the diet with 10% crude glycerin in the feeders (Cerrate et al., 2006). However, Barteczko and Kaminski (1999) experienced a different outcome in birds fed diets containing 10% crude glycerol, which showed improved broiler performance. Additionally, Simon et al. (1997) reported no effects on broiler chickens fed diets including up to 10% crude glycerol in a low-protein diet on N retention; however, in an earlier experiment by Simon et al. (1996) using a protein rich diet, N retention was found to be positively correlated with glycerol level in the diet up to 20%. Supporting this finding, Lammers et al. (2007), reported that egg production, egg weight, egg mass, and feed intake were not affected when crude glycerol was included in diets in up to 15% concentration for hens. Turkey hens fed glycerol as an energy source also showed no negative effect on laying rate, egg weight, and feed conversion (Rosenbrough et al., 1980). Additionally, laying hens consuming diets containing 2, 4, or 6% crude glycerin exhibited no significant effects on performance ($P > .05$) or on N, Ca and P balances (Swiatkiewicz et al. 2008).

Fewer studies have reviewed the effects of crude glycerol in ruminant diets. Donkin et al. (2009) determined crude glycerol up to 15% of the DM ration could replace corn grain in feeding rations. Crude glycerol also served as a suitable replacement for corn grain in dairy cows at inclusion levels of at least 15% DM without adverse effects on milk production or milk composition. The higher levels of crude glycerol (10-15%) resulted in more weight than did the

cattle fed rations including 0 or 5% glycerol. Moreover, body condition scores did not change with the different treatments. Schroder and Sudekum (1999) reported that diets containing 10% crude glycerin in dairy rations, effectively replaced over one-half of the starch in the diet while causing no significant effects in feed intake, rumen microbial synthesis, ruminal digestibility, or total-tract nutrient digestibility in steers. DeFrain et al. (2004) reported no negative effects on milk production and feed intake and that 5.4% of crude glycerol can be included in cow rations 14d to 21d before calving. These studies collectively indicate that crude glycerol can replace corn grain in lactating and dairy cattle rations.

DDGS

As previously stated, distiller's grains have been successfully implemented in cattle diets for decades. More recently, the feedstuff has been gaining interest for swine and poultry diets as a beneficial replacement as a protein and energy source (Morrison, 2007). Previous studies have indicated that DDGS provide lysine, phosphorus, and energy, and replaces soybean meal, dicalcium phosphate, and corn. Approximately 90.9 kg of DDGS and 1.36 kg of limestone can replace 80.9 kg of corn, 8.6 kg of 46% protein soybean meal, and 2.7 kg of dicalcium phosphate in a ton of complete feed for swine (Shurson, 2001). Ultimately, the nutritional value of distiller's grains has been evaluated in poultry (Wang et al., 2007; Lumpkins et al., 2005; Swiatkiewicz and Korelski, 2008), swine (Woerman et al., 2008; Xu et al., 2010) and ruminants (Leupp et al., 2009; Klopffeststein, 1974; Ham et al., 1994).

Wang et al., (2007) determined that chicks can be fed up to 25% DDGS without causing any adverse affects on the growth rate; however, the chicks fed the 25% DDGS experienced a reduction in feed conversion. Additionally, these results indicated that good quality DDGS could be used in broiler diets at levels of 15-20% with little adverse effect on live performance but

some possible loss of dressing percentage or breast meat yield. Swiatkiewicz and Korelski (2008b) reported that DDGS obtained from modern ethanol plants is an acceptable ingredient of poultry diets and can be safely fed at 5-8% in starter diets for broilers and turkeys, and 12-15% in grower-finisher diets for broilers and turkeys and also for laying hens. Lumpkins et al. (2004) formulated isocaloric and isonitrogenous starter, grower, and finisher diets ranging from 0-18% DDGS inclusion levels. This research showed no differences in performance and carcass yield except for a decrease in body weight gain and feed conversion during the starter period, when chicks were fed an 18% DDGS diet. Lumpkins et al. (2005) found no significant differences in the majority of egg production and egg quality measures between hens fed the diet with 0 or 15% DDGS obtained from modern ethanol plants; therefore, the authors concluded that DDGS was an acceptable feed ingredient for laying hens and could be successfully used in commercial layer diets at a 10-12% inclusion rate.

Woerman and Tilstra (2008) reported that DDGS are considered a valuable source of energy, phosphorus, lysine, and other nutrients for swine and can be included in grower finisher diets ranging from 5% to 20% and in sow gestation diets ranging from 10% to 40%. DDGS clearly can replace corn, soybean meal, and phosphorus but should be balanced using available amino acid values and available phosphorus so protein and phosphorus aren't overfed. Cromwell and Stahly (1986) reported up to 20% DDGS can be included, on an isolysine basis, in diets for growing-finishing swine with only a slight (3-4%) reduction in growth rate and efficiency of feed utilization. Xu et al. (2010) reported increasing the dietary DDGS content had no effect on ADG, but ADFI was linearly reduced, and G:F was linearly increased ($P>0.01$). No negative effects on growth performance or dressing percentage occurred when growing-finishing pigs were fed diets

containing up to 30% DDGS, but in diets including more than 20% DDGS, fat quality may be reduced.

Traditionally, distiller's feeds have been fed to ruminant species for a number of years and have proven a very valuable protein source; specifically approximately, 82% of DDGS are fed to cattle. Feeding DDGS in place of corn grain provides a valuable source of energy in the form of fermentable fibers. Also, neutral detergent fibers (NDF) and acid detergent fibers (NDF) are digested at a slower rate than other forms of energy such as starch, which can reduce the occurrence of acidosis in ruminants (Klopfenstein et al. 2001). Leupp et al. (2009) reported that feeding 30% DDGS did not affect any performance or carcass characteristics but did influence steak sensory attributes and color in growing and finishing steers. A study conducted by Ham et al., (1994) reviewed the comparison between wet distillers and dried distillers grains with solubles on ruminants. In the finishing trial, cattle fed the wet and dried distillers byproducts gained faster and more efficiently ($P < .05$) than cattle fed the dry rolled corn control diet. Although gains were similar ($P > .10$), cattle fed WDB consumed less feed ($P < .05$) and were more efficient ($P < .05$) than cattle fed DDGS. Obviously, distillers byproducts are a good source of bypass protein, and drying seems to have little effect on the value of the protein for growing calves. The growing trial data indicate distillers byproducts can be fed wet or dry for, equal protein value (Waller et al., 1994).

Overall, several previous studies have indicated that crude glycerol and DDGS are utilizable as energy sources in animal diets. The ubiquitous availability of both ingredients has allowed for a lower cost substitution of corn or soybean meal for monogastrics and ruminants; however, establishing an ingredient nutritionally is only one part in determining whether the ingredient can be economically feasible. For instance, variability has been shown to exist among

processing facilities. Because both ingredients are by-products from the biofuel industry, variability exists between facilities based off of the processing method and feedstock used; therefore, understanding the exact composition of each feedstock is critical in animal feed production to ensure safe feeding practices.

Variability

Glycerol

Biodiesel is produced using either vegetable oil or animal fat, which is first filtered to remove water and contaminants. Next, during transesterification, oils of varying purity levels are used to react with an aliphatic alcohol, typically methanol, to increase yield. Unfortunately, any residual methanol results in crude glycerol and can be toxic in levels exceeding 0.015%. Therefore, according to the FDA, methanol content in animal diets cannot exceed 150 ppm (0.015%). Specifically, methanol toxicity has the potential to cause severe damage to the central nervous system, vomiting, severe metabolic acidosis, blindness, and Parkinsonian-like motor disease in animals (Kerr, 2007). In addition, crude glycerol can contain a large number of other impurities such as spent catalysts, salts after neutralization, methyl esters, oil/fat, soap, and free fatty acids (Dasari, 2007). Most recently, interest in understanding the degree of variability that exists among feedstocks, processing methods, and techniques is imperative in determining crude glycerol a safe and viable option for the feed industry.

Initially, glycerol used in industrial applications requires an expensive purification process to remove impurities for uses such as cosmetic and pharmaceutical purposes. While purified glycerol is too expensive for use in the animal feed industry, a study conducted to evaluate the difference between technical and pure forms of glycerol on feeding values in pigs. Kijora et al., (2006) reported no significant difference on feeding performance in finishing swine.

Purifying the ingredient did not improve nutritional consequences in any other way than by removing the very minimal methanol content that has been shown to have no effect on animal performance in levels below 115 ppm or .015% (Kerr et al., 2007).

DDGS

During yeast fermentation of cereal grain, nearly all the starch is converted into ethanol and CO₂. The resulting DDGS contain 2-3 times more concentrated nutrients and have been recognized as having the ability to contain high crude protein, amino acids, phosphorus, and other nutrients; however, the main problem with utilizing DDGS is the high variability of nutrient composition and quality among different DDGS sources (Swiatkiewicz et. al., 2008b). In the past several years, a number of studies have evaluated the degree of variability that exists among DDGS depending on the feedstock source used, amount of solubles added back into the grain, and the duration of fermentation.

For example, Cromwell et al. (1993) reviewed the physical, chemical, and nutritional characteristics of distillers grains intended for swine and poultry consumption. Ranging from beverages to fuel alcohol production systems, nine sources were evaluated on variability of resulting DDGS. Results showed crude protein varied from 23.4 to 38.7%, NDF from 28.8 to 40.3%; ADF from 10.3 to 18.1%; fat from 2.9 to 12.8%; ash from 3.4 to 7.3%; lysine from 0.43 to 0.89%; methionine from 0.44 to 0.55%; threonine from 0.89 to 1.16%; and tryptophan from 0.16 to 0.23%. Color scores of the DDGS ranged from very light to very dark, and odor scores varied from normal to burnt. Lysine content was reported to be lowest when darkest and showed significant correlation with the lysine content. Also, ADF concentration showed a tendency to negatively correlate with the nutrition value and digestibility of DDGS.

In 2002, Spiels et al. were able to determine an average nutritional content of 118 corn DDGS samples originating from 10 new ethanol plants. Average proximate analysis concluded crude protein to be 30.2%; crude fat 10.9%; crude fiber 8.8%; ash 5.8%; NFE 45.5%; ADF 16.2%; NDF 42.1%; lysine 0.85%; methionine 0.55%; calcium 0.06%; and phosphorus 0.89%. Results indicated that the highest amount of variability among sources existed especially for lysine and methionine concentrations, and crude protein, crude fat, lysine, methionine, threonine, and phosphorus levels were higher in comparison to reference data (NRC, 1994). In addition, Belyea et al. (2004) determined that high variation in DDGS from multiple production facilities was not related to the composition of corn used in fermentation but directed more towards the processing techniques used. For example, drying temperature and duration, amount of solubles added back, original grain composition, and efficiency of starch fermentation during ethanol production were specific factors.

Handling/Storage/Transportation

New ingredients introduced into the animal feed industry pose a significant concern about the ability to handle, store, and transport such materials effectively. Materials that are difficult to handle generally require more labor and processing steps that can offset the cost-effective benefits of utilizing a cheap and available ingredient. In particular, the physical make-up of the material can either impair or improve the functionality of the storage and handling characteristics. Therefore, predicting the flow ability in a feed mill system requires the consideration of a number of factors; bulk density, viscosity, pelletability, and moisture content can each impair the ease of flow through the system. Each of these factors contributes to feed mill flow and plays a critical role in reducing labor, equipment, and transportation costs. Presently, crude glycerol and DDGS are considerably new to the feed industry, and few studies

have been conducted to evaluate the behavior of each ingredient throughout handling, storage, and transportation.

Flow characteristics are traditionally predicted by knowing the viscosity in liquids or the bulk density in solid materials. For example, glycerol is described as being a very viscous, water soluble liquid that tends to thicken in low temperature environments, indicating potential problems with flow properties through the system depending on temperature. Additionally, at lower temperatures, glycerol may form crystals that melt at 17.9° C, but does not freeze (Pagliaro et al. 2008). Recently, researchers reported that adding small amounts of water incrementally alters the physical makeup of crude glycerol so it becomes a thinner, freer flowing liquid (Johnson, 2010).

Bulk density is used to distinguish flow ability of dried materials. Several studies have validated that DDGS are historically known for having a low bulk density, which causes problematic material handling characteristic. The low bulk density impairs flow characteristics and reduces bin space. Additionally, DDGS have a flat or pleated like particle shape and tend to stack causing the material to cake or bridge in bins and trucks. Studies have been conducted to improve DDGS flow characteristics by pelleting the material.

Next, concerning storage, the difference between storage characteristics for dry versus liquid materials is huge. First, liquid ingredients tend to be perishable and require specialized storage to prevent aerobic decay. Options such as sheeting, sealing, and short-term storage can prevent aerobic micro-organisms from flourishing. Also, viscous liquids require a positive displacement pump in the bin to effectively transfer the material through the system. One important aspect to consider when looking for storage options is the acidity of the material; galvanized storage bins are not suitable for acidic ingredients due to the zinc in the steel

dissolving and potentially causing zinc toxicity. Furthermore, since crude glycerol has the propensity to separate into layers during storage, mixing the glycerol in-silo would be of considerable value to assure uniform composition (Crawshaw, 2001).

Fortunately, crude glycerol has shown no indication of spoilage during short-term storage. Notably, Sudekum and Schroeder (2008) reported that in a pelleting study, crude glycerol improved storage characteristics. Three different purity levels of crude glycerol were reviewed for physical, chemical, and hygiene pellet quality characteristics. Concentrates were stored under ideal (15 C, 60% RH) or poor (20 C, 70% RH) environmental conditions for a duration of 4 or 8 weeks. The physical quality of the pellets was not affected with regards to purity or concentration of glycerol, and the glycerol seemed to improve hygiene properties of the pellet without reducing the quality. Even at low concentrations (50g/kg DM), glycerol had a preserving effect, possibly due to the hygroscopicity of glycerol, which decreases water activity, reducing the water available to undesirable microorganisms in pellets (Heiss et al., 1994). Indeed, this preserving effect of glycerol has become widely sought in the food industry. In regards to the feed industry, Johnson (2010) included that adding crude glycerol did not appear to cake in the mixer or feed bunks, and it actually serves as a coating agent to the feed, reducing the amount of dust.

Second, dried ingredients tend to be more predictable based on the materials' anticipated bulk density and moisture content. For instance, DDGS are generally dried to 10-12% moisture content to eliminate possible spoil during storage. However, DDGS require more attention to transportation characteristics of the dry ingredient. Since DDGS have a lower bulk density, the rail cars fill up before they reach their maximum weight allowance, reducing overall transportation efficiency. Additionally, load in/out times are affected when the material doesn't

flow properly, and such material also requires more manual labor, increasing labor costs. An attempt to resolve this issue has been to pellet the DDGS, which has proven to improve bulk density, allowing more material to be transported per rail car, decreasing the cost of transportation, and improving flow characteristics.

Rosentrater and Kongar (2009) reviewed the cost factor of implementing pelleting technology within the ethanol processing facility to improve transportation and flow ability of DDGS. They reported that with increasing DDGS generation rates, the cost of pelleting is significantly reduced because of the economies of scales. For instance, the cost to pellet 100 ton/d was \$14.07/ton/y, whereas at a heightened scale of 1000 ton/d, the cost to pellet was reduced to \$3.95/ton/y. Therefore, the option of including a pelleting line is appealing to the ethanol industry because the cost to pellet is minimal in large scale production facilities and the benefits are huge. Clearly, if ethanol facilities were to pelletize their DDGS, transportation and flow ability of the ingredient would greatly be improved.

For new ingredients that show poor handling properties such as crude glycerol or DDGS, pelleting may be the only option to utilize the ingredient. Consequently, a number of factors pertain throughout the production process to keep the ingredient economically feasible. Pelleting a diet allows for freer material flow in and out of trucks, which alleviates complications associated with bridging while allowing loading and unloading times to be kept to a minimum. Ultimately, however, when pelleting an ingredient to improve flow ability and handling characteristics, feed mill managers should consider all variables associated with manufacturing pellets to gain maximum pellet mill efficiency and pellet quality.

Pelleting

Operations

Pelleted diets in comparison to mash diets have long been understood to improve bulk density, resulting in increased storage and transportation capacities without compromising the nutritional value. Pelleted diets also improve feed efficiencies by reducing segregation of ingredients, minimizing natural losses from wind or spillage and enabling the feed ingredients to be gelatinized with a combination of moisture, heat, and pressure, allowing for better utilization by animals (Thomas, 2005).

To process feed, one of the main considerations is cost. Traditionally, if the benefits can outweigh the costs, then the method of production is justified. Yet, the pelleting process is considered the most expensive production method in the feed mill in terms of equipment cost, energy cost and capital investment; however, the improved handling characteristics and feed efficiencies will generally offset the additional costs. Specifically, electrical energy usage is significant in the pellet mill, feeders, conditioner and the cooling system, and make up the majority of the cost required in pelleting. Furthermore, steam is incorporated in the pelleting process to condition the material to improve pellet quality and pellet mill throughput. In addition to costs involved in operating the pellet mill, overhead costs such as maintenance, equipment replacement and repair must be considered and can be variable depending on frequency and type.

Operating the pelleting system at the maximum production rate possible improves energy efficiency, reducing overall kilowatt-hours per ton (kWh/ton). Motor load and diet formulation are two main contributors to reducing energy consumption. For instance, motor load is the actual operating amperage of the pellet mill motor divided by its top rated amperage capacity, providing a percent at which the pellet mill is operating. Maximum motor load produces the

greatest production efficiencies because reduced mechanical energy is required at the die. Type of diet is another factor in reducing energy costs because different formulations have different frictional behaviors varying mechanical energy requirements (Garrison, 2005).

A number of parameters have been considered to maximize pellet mill efficiency while aiming for the best pellet quality possible. Understanding the cost of production, as stated above, is of primary importance in improving production efficiencies; however, the parameters for evaluating pellet quality additionally affect production efficiencies and can be examined accordingly. According to Behnke (1994), pellet quality has been primarily broken down into five main components: formulation (40%), fineness of grind (20%), steam conditioning (20%), die selection (15%) and cooling/drying (5%).

Knowing the complete composition of the ingredients of the diet being pelleted is very important in determining optimum production capabilities and pellet quality. All ingredients generally contain different levels of protein, fat, fiber, starch, density, texture, and moisture, and each uniquely impacts the pelleting process. Fortunately, a pelletability chart is available that provides information for understanding pelleting characteristics of a number of common ingredients. Since such information stipulates pelletability and abrasiveness of ingredients, feed formulation and production rates can be estimated. For determining pellet quality based on formulation, fat and protein content have shown the most effect. Briggs et al. (1999) and Winowiski (1988) and are in agreement that increasing protein content increases pellet durability. In addition, Briggs et al., (1999) reported in the same study that increasing the oil content resulted in a negative effect on pellet quality.

Previous studies have indicated that a variety of particle sizes in a ration are desirable to aid in pellet quality and production rates. As particle size slightly varies within a diet, air voids

within the pellet are eliminated, which offered increased contact surface area. This gives the material the ability to accept a more thorough steam conditioning, producing a more stable pellet. A study conducted by Stevens (1987) determined that pellet mill efficiency decreased with decreasing particle size by up to 12% in a 72.4% ground corn and 20% ground soybean meal ration as well as in a 72.4% wheat and 20% soybean meal ration. Since in both the corn and wheat based diets the kWh/ton decreased by about one unit per ton between coarse and fine ground grain, depending on the amount produced, that difference could substantially decrease the amount of energy used. When considering pellet quality with respect to particle size, Wondra et al., (1995) reported that particle sizes of 600 microns of corn, or slightly less, seemed to be optimal for finishing pigs.

Mash conditioning is described as the amount of heat and moisture introduced to the diet, and it should be completely understood to keep production rates optimal and feed mill inefficiencies minimal. Proper conditioning of the meal is essential because different types of feed require varying amounts of moisture and heat. For example, some feeds have the ability to caramelize at lower than normal temperatures and will cause pellet die plugging. To account for this, the pelletability chart offers general guidelines for suggested temperature and moisture to be added per type of ingredient, which is endorsed in the literature. In addition to optimizing mill efficiencies and production rate, mash conditioning also contributes to pellet quality and die and roll life (Garrison, 2005).

In addition, the physical characteristics of the pelleted formula is important to feed mill efficiency, multiple factors within the pellet mill itself can be easily overlooked. The pellet mill consists of rolls and a die. Each roll forms a nip between itself and the die, which is where the actual pellet is formed. The roll can be adjusted, and the space between the roll and die can vary.

However, if the roll is adjusted too tightly against the die, too much metal to metal contact will occur and so will die peening, causing poor production rates and plugging. To avoid these complications, roll gap is most commonly controlled automatically and is set according to the diet being pelleted. According to the American Feed Industry Association, pellet quality is significantly improved with a wider roll-gap setting, but starting the pellet mill with a wide roll gap is difficult. In this case, automatic adjusters are used to narrow the gap at start up and then move out to a wider gap throughout pelleting.

The L/d ratio, which is the performance ratio and is defined as the relationship between the effective length (L) divided by the pellet diameter (d), has been shown to have an impact on pelleting efficiency. High L/d ratios provide high pellet/die resistance and a longer amount of time in the die hole (die retention) to be able to bind and form a pellet. Meanwhile, low L/d ratios cause lower resistance and less time in the die hole. Each ingredient has a specific L/d ratio requirement to form the material in a pellet (Garrison, 2005). Generally, as the L/d ratio decreases, an increase in production capacity and decrease in pellet quality is experienced.

The cooling and drying process after pellets are formed contributes approximately 5% of the relative efforts to gain the best pellet quality possible. According to Alles (2005), the cooling process can add or subtract as much as 7% to or from the quality of the pellet. Pellet durability, affected by cooling/drying for evaporative-type cooling, has been established as maximizing pellet quality when hot air is moved across hot pellets, warm air is moved across warm pellets, and cool air is moved across cool pellets.

Nutrition

Pelleting improves animal nutrition by ensuring a balanced ration is consumed by eliminating the chance of ingredient segregation and improving nutrient delivery and efficiency.

During mash conditioning, moisture, heat, and pressure are introduced to gelatinize or break down components of the diet, improving nutrient utilization and growth performance in swine and poultry. Additionally, pelleting reduces waste from spillage or wind. These benefits combined greatly justify the extra costs associated with pelleting animal feed. Traditionally, growth and performance is analyzed by reviewing the average daily gain (ADG), feed to gain ratio (G/F) and average daily feed intake (ADFI). Previous studies have indicated that livestock receiving pelleted diets generally have higher average daily gains and lower feed conversion compared to mash diets in poultry (Amerah et al., 2007; Hamilton and Proudfoot, 1995) and swine (Ohh, 1991; Chae et al., 1997).

According to Benhke (1994), benefits from pelleting feeds for broilers are attributed to the thermal modification of starch and protein, improved palatability and decreases in feed waste selective feeding, ingredient segregation, less time and energy spent in prehension, and fewer pathogenic organisms. Amerah et al., 2007 found that broiler performance increased with pelleted diets compared to mash diets ($P < 0.05$); however, they found that pelleted diets resulted in negative effects on metabolizable energy ($P < 0.05$). Hamilton and Proudfoot (1995) evaluated the effects of pellet diets on the performance of broiler chickens versus either coarse or fine mash. Both diets improved ($P < .0001$) 21 day and 42 day body weights, feed conversions, and economic returns for feed and chick costs. However, the researchers concluded that improved broiler productive performance could be due to an increase in daily nutrient intakes and digestibility of dietary carbohydrates associated with pellet form.

Pelleted diets have additionally been proven effective in swine diets. Improved feed efficiency, ADG, and energy availability were experienced with such feed versus a mash diet (Ohh, 1983; Chae et al., 1997). NCR-42 Committee on Swine Nutrition in 1969 reported that

pelleting improved ($P > .05$) feed conversion and had no effect on ADG in a cooperative study to compare corn-soybean meal diets in pellet and mash form. Back then, researchers concluded that the cost of producing pelleted feed could not be justified economically. However, over the years technological advances have improved production efficiencies, and the benefits of pelleted diets have proven to be economically feasible. Later, Ohh (1983) summarized the effects of pelleted versus mash diet in swine and showed an improvement of 3-4% in growth performance and feed efficiency in swine from 16 trials. In agreement, Chae et al., (1997) showed an average daily gain of 20-90 kg of body weight in pigs fed pelleted diets. Both experiments noted an improved feed conversion rate in pelleted diets over that of mash feeds.

The benefits of feeding a pelleted diet have been established for a number of years and proven effective in both swine and poultry. Groesbeck et. al., 2009 concluded that diets containing crude glycerol, pellet quality and production efficiencies improved. Increasing crude glycerol increased both the standard (linear and quadratic, $P < 0.01$) and modified (linear, $P < 0.01$; quadratic, $P \leq 0.02$) pellet durability indexes up to 9% with no further benefit thereafter. Notable, adding crude glycerol decreased (linear; $P < 0.01$) production rate (t/h) and production efficiency (kWh/t). Evidently, crude glycerol has shown to improve pellet quality while providing a cheap energy source for both poultry and swine.

In 2008, Fahrenholz et al. conducted a study pelleting a varying level of DDGS to review pellet quality and pellet mill efficiency. DDGS were replaced for corn in up to 40% inclusion level. There was no significance in pellet quality on the level of DDGS. Production rate experienced a 10% lower production rate with the inclusion of 40% DDGS and a statistically significant difference in electrical consumption in diets including 10% DDGS ($P > .04$). There was a significantly lower mash bulk density ($P > .005$) with 30 and 40% DDGS differing from the

control, and pellet bulk density ($P > .0001$) with all levels differing from the control. Overall, the inclusion of DDGS had the most effect on production rate and bulk density of both mash and finished pellets due to the initial bulk density of DDGS.

Groesbeck et al. (2008) and Fahrenholz et al. (2008) findings have contributed to the motive of the following experiments included in this thesis. Being able to utilize both crude glycerol and DDGS in the animal feed industry will provide an alternative option for replacement energy sources. Therefore, it was our intention to improve pellet quality, operational characteristics, flow properties, and nutritional consequences in the manufacturing animal feed diets including DDGS and crude glycerol.

Chapter 1 - Storage and Variability Considerations of Crude Glycerol

Introduction

When contemplating the use of a new ingredient to be included in an animal diet, multiple factors are involved in determining the cost effectiveness of the material. Aside from manufacturing considerations, other variables should be reviewed in order to keep cost of production low. Previous studies have indicated that variability exists between the chemical composition of crude glycerol from various sources, indicating potential limitations on storage, handling and inclusion levels could arise. Hansen et al. (2009) reported that the chemical composition of the crude glycerol samples from seven Australian biodiesel manufacturers varied considerably; glycerol content ranged between 38 and 96%, with some samples containing up to 29% ash and 14% methanol. Contrarily, Johnson et al. (2006) reported that there was very little variation in the chemical and physical properties of crude glycerol from seven different vegetable oils. Little information is available on the chemical composition of crude glycerol from vegetable oil sources in comparison to animal oil sources; therefore, experiment 1 reviewed the chemical composition of crude glycerol from vegetable and animal oil sources and the degree of variability that exists.

In addition to variability, storage and handling are other factors that must be considered when implementing a new ingredient in the animal feed industry. Crude glycerol is a very viscous material and has poor flow properties, especially during low temperatures causing potential issues with handling. Piping and holding bins material should be tested so the ingredient doesn't cause corrosion or other deterioration. There is limited information on the

ability to store and handle glycerol; therefore, for exp. 2, we reviewed the effects of crude glycerol on two different types of metal in efforts to further understand the storage capabilities.

Materials and Methods

Exp. 1 Variability Study

For experiment one, thirteen U.S. biodiesel production facilities were solicited for a survey to quantify the biodiesel industry and determine the degree of variability between feedstock sources and resultant crude glycerol. Physical and chemical properties of crude glycerol originating from individual biodiesel production facilities over time and across multiple biodiesel production facilities were quantified. Each facility provided representative samples of the feedstock (i.e. animal fat, soybean oil, corn oil) and resulting glycerol. All samples collected were characterized for color, viscosity, particulate matter, pH, moisture content, methane, fat content, and gross energy at SDK Laboratories, Hutchinson, KS.

The tests conducted were chosen based on previous research. Hansen et al., (2009) reviewed results of feedstocks and glycerol composition based on a set of parameters that would provide evidence of variability. During biodiesel production, methanol is used to transesterify the triglycerides, leaving some methanol residue. In order to assure feeding standards, an analysis on the amount of methane present in the glycerol was conducted. Moisture content was measured using the Karl Fisher method, which is a volumetric titration used to determine trace amounts of water in a sample and served as a measurement of moisture content. Residual fat content remaining was tested by acid hydrolysis to determine the yield ratio of fat utilized in biodiesel production. A bomb calorimetry method was used to test the overall gross energy by measuring the heating value of the substance. Particulate matter was analyzed by the amount of insoluble impurities (AOCS Ca 3a-46 standards). To determine fluid flowability of the glycerol, viscosity

was tested at 4.45°C which reflects storability characteristics and feed mill flow. The Gardner color method was used to determine the degree of impurity level in both vegetable and animal variation of glycerol. Finally, pH was tested to determine the acidity of the glycerol, which would help determine a corrosion level of the glycerol. These tests assist in determining the implications for the type and inclusion level of the crude glycerol depending on feedstock source.

Exp. 2 Storability Study

Experiment 2 was conducted to evaluate the storability of glycerol under room temperature and excessive heat conditions. The experimental design of this study consisted of two environmental chambers in which mimicked room temperature conditions (23°C and 50% relative humidity) and excessive heat conditions (60°C and 5% relative humidity). Twelve 1.5 L plastic containers were filled with crude glycerol obtained from Prairie Pride, Inc. a biodiesel plant in Deerfield, MO that produces biodiesel from 100% soybean oil. Stainless steel or ferrous metal strips were submerged in each of the containers and the containers were placed in the environmental chambers. Visual observations were recorded weekly over a period of two months.

For experiment 2, containers with metal were analyzed in triplicates. The majority of bins and silos used in the grain industry is a ferrous metal sub-straight with protective zinc coating, which allows discoloration but refrains from corrosive activity; however, understanding the behavior of a new ingredient on handling and storage will determine the bin and piping material needed to withhold the substance. The glycerol in this study was provided by Prairie Pride Inc. and stored in heated agitated stainless steel tank. The main objective of this study was to

determine if crude glycerol can be stored and handled under common feed mill storage operations.

Data Collection

The chemical and physical properties of the feedstock and glycerol samples assayed are presented in Table 1. Each sample was analyzed at SDK Laboratories in Hutchinson, KS in duplicates. Their testing procedures and methods are provided in Table 1-2. Results were then compiled and put into a statistical analysis software program to review variability. For experiment 2, visual observations were made weekly over a two month period. Temperature and relative humidity were recorded and held constant over the duration of the experiment.

Statistical Analysis

Statistical analysis was performed using mixed procedures (SAS Inst. Inc., Cary, NC). The results from experiment 1 provided data that was analyzed through SAS. Contrasts for linear and quadratic polynomial effects of glycerol based on variability were also included in the analysis. In experiment 2, visual observations and photographic evidence were used to explain the corrosiveness of crude glycerol on stainless steel and ferrous metals.

Results

Exp. 1

The chemical and physical properties of the feedstock and glycerol samples assayed are presented in Table 2.1. There was considerable variation between samples, particularly when comparing glycerol from the different feedstocks (vegetable vs. animal). As expected, the gross energy content was higher in the starting material compared to the glycerol, as the fatty acids are removed during the production of biodiesel. There was more residual fat remaining in the

glycerol from animal fat, and the residual methanol was consistently higher compared to that from vegetable oil because of the free fatty acid composition of animal tallow prior to biodiesel production. As both fat content and particulate matter increased, clarity and viscosity decrease. The color of the samples varied between vegetable and animal sources is most likely due to the difference in pigment and other components within the feedstock.

These results agree with Hansen et al., (2009) that variability exists between biodiesel facilities depending on feedstock source and method used. Crude glycerol has the potential for being included in animal diets by supplying an economically feasible energy source; however, levels of residues, such as particulate matter and methanol, and other parameters including pH, moisture, fat, viscosity, etc. in the crude glycerol need to be monitored. By doing this, it will help prevent excessive amounts of these compounds entering into animal diets. Additionally, testing requirements and frequency of the crude glycerol need to be determined to comply with ensuring the ingredient fit for animal consumption at all times.

Exp. 2

There was no notable corrosion during the two month experiment for either metal type under the two environmental conditions. There was a slight discoloration observed on the ferrous metal stored under summer conditions, but no pitting or sign of corrosion was noted. No changes were observed in the stainless steel under either environment condition, indicating a better material option for long term storage for crude glycerol. Even though stainless steel might be required to store crude glycerol, the higher cost factor of stainless is still an issue in keeping the cost of crude glycerol as a feed ingredient low.

Discussion

The results of this experiment have validated a difference in chemical composition of crude glycerol from varying feedstocks. The importance of understanding the chemical composition is vital when implementing the substance in an animal diet because serious repercussions can arise from feeding an unsafe ingredient. As previously stated, methanol content can have a detrimental effect on animals when not tested for. Current inclusion levels of crude glycerol in feed are low; therefore, regardless of variability, as long as the methanol content is within feeding range the substance can be utilized. Based on the results from exp. 1, it can be presumed that variability does exist in glycerol from varying feedstocks used for biodiesel production. Therefore, the ingredient should be examined for proximate analysis before using to ensure a safe and nutritional feeding value.

For exp. 2, the stainless steel used in this experiment was 304L austenitic alloy, which is low carbon metal containing 18-19% chromium. The chromium in the alloy provides a chromium oxide protective layer, which serves as a resistance to corrosion on a wide range of moderately oxidizing to moderately reducing environments. Unfortunately, stainless steel is not commonly found in feed mills due to the cost associated with the highly versatile metal. Therefore, the stainless metal was compared to a high carbon ferrous metal that is commonly found in feed mill facilities. The amount of carbon in metals influences the degree and rate that oxidation occurs, potentially leading to rust and corrosion. Indeed, the first sign of oxidation is discoloration. It was evident in this experiment that the ferrous metal versus the stainless steel contained a higher amount of carbon. Ferrous metal corrodes at atmospheric conditions and is commonly coated with a preventative layer such as zinc to reduce the prevalence of corrosive behavior. Although the ferrous metal appears to be affected by the glycerol, discoloration would

occur at normal atmospheric conditions. Therefore, since crude glycerol is a non-acidic, natural product it can be presumed that the presence of glycerol did not serve as an enhancing agent to the degree of discoloration of the ferrous metal over two months.

Implications

Variability in an ingredient used in the animal feed industry is definitely not ideal and can pose a threat to animal performance; however, with further experimentation, variability between feedstock sources and processing methods can potentially be defined and the threat could be eliminated. In addition, the storage capabilities of a material should be completely understood prior to usage to ensure safe feeding and quality control. The provider of the crude glycerol used in this experiment stored their glycerol in heated, agitated stainless steel tanks to refrain from ingredient separation, thickening and bin deterioration.

Figure 1.1 Corrosive activity from crude glycerol on stainless steel

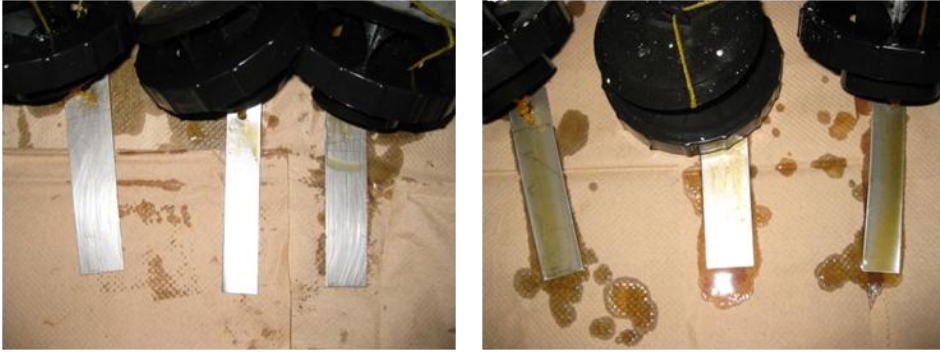


Figure 1. The effects of glycerol on stainless steel (a) initial and (b) final after two months of excessive heat conditions.

Figure 1.2 Corrosive activity from crude glycerol on ferrous metal

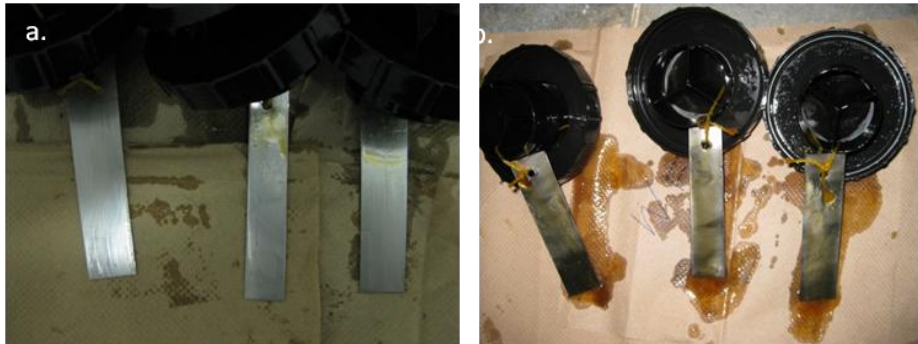


Figure 1. The effects of glycerol on ferrous metal (a) initial and (b) final after two months of excessive heat conditions .

Figure 1.3 Corrosive activity from crude glycerol on stainless and ferrous metals, exp. 2.

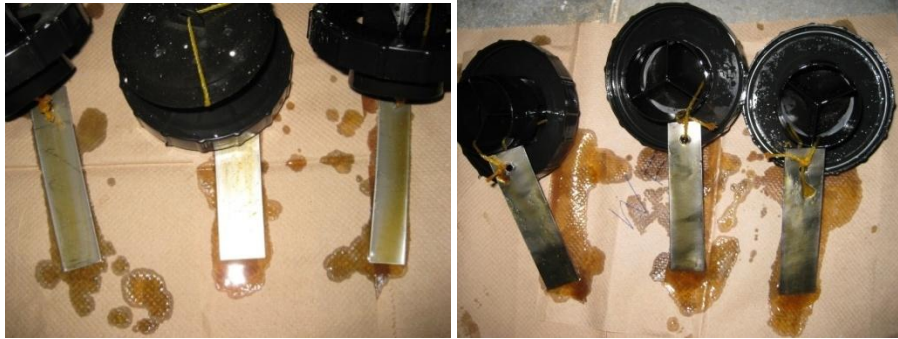


Figure 1. The effects of glycerol on stainless steel (a) and ferrous metal (b) after two months of excessive heat conditions .

Table 1.1 Composition of glycerol obtained from commercial biodiesel production facilities

Variable	Vegetable		Animal	
	Feedstock	Glycerol	Feedstock	Glycerol
Dry Matter, %	100 (\pm 0.03)	90.3 (\pm 1.05)	99.5 (\pm 0.33)	85.0 (\pm 7.1)
Fat, %	93 (\pm 1.27)	0.387 (\pm 0.07)	94.5 (\pm 0.82)	15 (\pm 7.5)
pH	5.9 (\pm 0.29)	6.53 (\pm 0.23)	7.04 (\pm 1.07)	8.47 (\pm 0.94)
Methanol, ppm	0.0 (\pm 0)	8.99 (\pm 4.1)	40.3 (\pm 29.3)	470 (\pm 232)
Particulate matter, ppm	6.6 (\pm 5.3)	0.9 (\pm 0.65)	6.8 (\pm 4.6)	8.4 (\pm 7.7)
Viscosity, cSt/s	79 (\pm 49.2)	82 (\pm 19.3)	82 (\pm 43.0)	38 (\pm 15)
Gross Energy, BTU/lb	15,757 (\pm 80.5)	6,005 (\pm 109)	15,791 (\pm 100)	7,482 (\pm 1,005)
Color, s.u.	7.5 (\pm 2.2)	3.5 (\pm 0.9)	11 (\pm 1.68)	11 (\pm 1.9)

Table 1.2 Analytical Testing Methods SDK Laboratories, Hutchinson, KS

Test	Method	Official Method	Description
Viscosity	Viscosity	ASTM D445	Determines kinematic viscosity
Energy	Bomb	ASTM D240	Determines the heat of combustion of liquid hydrocarbon fuels ranging in volatility from that of light distillates to that of residual fuels.
	Calorimetry		Determines color by comparison with standards of specified color.
Color	Gardner Color	AOCS Td 1a-64	
Moisture	Karl Fisher	AOAC 966.20 16th Ed., 1995	Volumetric titration used to determine trace amounts of water in a sample.
		AOAC 954.02, 16th Ed., 1995	
Fat	Acid Hydrolysis	Ed., 1995	Determines free fatty acid content.
	Gas	AOAC 973.23, 16th Ed., 1995	Determines methanol in crude oils by multidimensional gas chromatography.
Methanol	Chromatographic	Ed., 1995	
Insoluble Impurities		AOAC Ca 3a-46	Determines dirt, meal and other foreign substances insoluble in kerosene and petroleum ether.

Chapter 2 - Utilizing Glycerol in Swine and Poultry Diets: Feed Manufacturing Considerations

Introduction

Previous research has indicated crude glycerol fit for animal consumption, providing the feed industry with a high-energy, available and cost effective ingredient; however, questions still arise in the manufacturing considerations of the material. Crude glycerol is a very viscous, water soluble liquid that has been only implemented as an animal feed ingredient in very few studies. Since it contains a unique composition, additional studies are required to validate and optimize production characteristics. Groesbeck et al., 2008 reported increasing glycerol concentrations increased (quadratic; $P>0.01$) pellet durability index through 9%, while decreasing (linear; $P>0.01$) production rate (t/h) and production energy (kWh/t).

Throughout the pelleting process, steam is introduced in the conditioner to gelatinize the starch in efforts to improve binding characteristics at the die. When the conditioning temperature increases, more steam and moisture is introduced into the pelleting system, reducing the viscosity of glycerol and potentially serving as a lubricant to the pellet die. Groesbeck et al., 2008 set the conditioning temperature constant at 150° F and only reviewed the effects of increasing concentrations of crude glycerol on pellet mill performance and pellet quality. In continuation of these results, we reviewed the effects of increasing crude glycerol concentrations as well as increasing conditioning temperature to determine if energy consumption and pellet quality would further improve.

Materials and Methods

Exp. 1 Pilot Study

Experiment one was conducted at the Feed Processing Research Center in the Department of Grain Science at Kansas State University on the effects of including crude glycerol on pellet mill efficiencies and pellet quality. Crude glycerol was obtained from Prairie Pride, Inc. a biodiesel plant in Deerfield, MO that produces biodiesel from 100% soybean oil. A 30 HP California Pellet Mill (Crawfordsville, IN) 1000 series “Master HD” model pellet equipped with a 3.97 mm (5/32”) x 31.75 mm (1 ¼”) (hole diameter vs. effective die thickness) pellet die. A Bliss Industries (Ponca City, OK) 30.5 cm (12”) by 121.9 cm (48”) steam conditioner was used to condition feed prior to pelleting. All treatments were conditioned with the same retention time, which was set using a variable frequency drive (VFD). The mash feeder screw was also set to a constant RPM for all treatments using a VFD. All diets were steam conditioned to 150, 170, and 190° F by adjusting the steam flow rate. Pellets were cooled using a double-pass perforated deck cooler (Wenger Manufacturing, Sabetha, KS). All experimental runs were performed using a warm pellet die.

For experiment one, a poultry finisher diet was formulated to contain 0, 3, 6 and 9% crude glycerol. Crude glycerol was substituted for only corn in the diet and no attention was given to the overall nutritional balance of the diet in this experiment to refrain from obstructing any effects from other ingredients on the pellet quality or pellet mill performance. The results for experiment one determined optimized processing conditions within our feed mill which lead us to a more precise prediction in estimating ideal crude glycerol inclusion levels and conditioning temperature for experiment two.

Exp. 2 Commercial Study

Experiment two was conducted at Don's Farm Supply, a commercial grade feed mill located in Newel, IA. The same crude glycerol obtained from Prairie Pride, Inc. was used in a turkey grower diet at the 3% level and was pelleted using a 500 HP Bliss Pellet Mill (Ponca City, OK). Steam conditioning and production rate was held constant. Samples of pellets were collected off the cooler and pellet mill amperage was recorded. The main objective of this study was to determine if a large batch diet could be pelleted when crude glycerol was included at a 3% concentration while maintaining ideal conditions and production efficiencies. Crude glycerol was substituted for only corn in the diet and no attention was given to the overall nutritional balance of the diet in this experiment to refrain from obstructing any effects from other ingredients on the pellet quality or pellet mill performance.

Data Collection

Exp. 1 Pilot Study

Pellet mill production data was collected on all diets (Table 1.1). Each diet run was replicated in triplicates by manufacturing a new batch of feed each time. Pellet mill electrical consumption (kWh/ton), production rate (t/hr), hot pellet temperature (°F) and pellet durability (%) were measured. Electrical energy data was collected using an Amprobe (Miami, FL) DMII (Pro) Data Logger Recorder, which read voltage and amperage data which was compiled to report pellet mill electrical consumption in kilowatt-hours per ton (kWh/ton). Production rate was measured directly at the pellet mill by collecting pellets at the discharge for 15 seconds, weighing and calculating production in kilograms per hour. Samples were taken consecutively three times at equal intervals and an average value was determined and used as the production rate for the run. Conditioning temperature was measured through a stiff thermocouple placed in

the steam of the conditioned mash as it moved from the conditioner to the pellet die and was adjusted by controlling the steam flow rate. To measure the hot pellet temperature, pellets were collected in a circular motion in an insulated foam pail, and the temperature was taken using a stiff thermocouple after the temperature equilibrated. Pellet quality was measured and reported as the pellet durability index (PDI) using the tumbling box procedure ASAE S269.4 (ASAE, 1991). The procedure requires the pellets to be evaluated in a standard and modified form after cooled. Two standard and two modified (inclusion of five 12.7 mm (1/2") hex nuts) PDI tests were conducted for each production run and an average value for each was reported.

Exp. 2 Commercial Study

Pellet mill production data was reported for experiment two. Three one ton batch diets were formulated to contain 3% crude glycerol. Production rate, conditioning temperature, pellet mill electrical consumption and pellet quality were the parameters of interest that were evaluated. DFS, Inc. facility consisted of state of the art equipment, control room and computer software systems allowing us to view the experiment from the control room as highly trained professionals made adjustments as needed throughout the runs. Amperage readings were recorded every half second throughout the entire run (Figure 1-1). Production rate and conditioning temperature were held constant once stable conditions were achieved. Pellet quality was tested using the same pellet durability index that was used in experiment one.

Statistical Analysis

Experiment one was run as a 3 x 4 factorial randomized complete block design (RCBD) consisting of four treatments containing 0, 3, 6, and 9% crude glycerol steam conditioned to 150, 170, and 190° F. Statistical analysis was performed using the general linear model (GLM) of SAS (v. 9.2). Analyses were completed for standard PDI, modified PDI and energy usage.

Contrasts for linear and quadratic polynomial effects of glycerol based on diet and conditioning temperature were also included in the analysis. Results were analyzed as an incomplete factorial design due to the inability to retain results from the 190° F conditioning temperature. Since there was not an interaction between the other variables measured, we had to look at the main effect to describe what happened. Qualitatively, a conclusion was drawn from reviewing the results on an individual basis. Experiment two was analyzed based off of visual observation. The general linear model of SAS (v. 9.2) was used to determine significance between pellet quality. Electrical consumption was analyzed best by qualitatively describing the main effect, which can be viewed in Figure 2.

Results

Exp. 1

Results of experiment one is presented in Table 1-3. Neither glycerol addition nor conditioning temperature significantly impacted electrical energy usage during pelleting. Pellet mill energy consumption is measured by recording motor load, amperage and voltage. There was less frictional heating across the face of the pellet die as conditioning temperature increased (linear; $P < .001$); however, glycerol did not impact hot pellet temperature. The decrease in pellet die friction indicates a decrease in motor load or the energy required by the pellet mill to rotate the pellet die.

Pellet quality increased ($P < 0.001$) as glycerol concentration and conditioning temperature increased. Though there was not a statistical interaction found between glycerol and conditioning temperature, the pellet mill consistently plugged with diets containing glycerol as the conditioning temperature increased. Out of three attempts, conditioning diets with 9% glycerol to 190°F plugged the pellet mill twice. Data from each of the conditioning temperatures were

obtained with diets containing 3 and 6% glycerol, as conditioning temperature increased, the pellet mill rolls began to slip. Diets containing 9% glycerol were reported as a failed experiment and statistically analyzed as an incomplete factorial design to illustrate an explanation from the main effect. Based upon these observations, it appears that the inclusion of glycerol in a diet improves feed quality, however, glycerol inclusion levels are limited to 3% or less and conditioning temperature is limited to 150° F due to the negative reaction with roll slip and mill plugging to occur.

Exp. 2

Results from experiment 2 were consistent with the first experiment, in that qualitatively there was an interaction between condition temperature and the addition of glycerol. The addition of glycerol improved ($P < 0.05$) pellet durability. Figure 1-1 and shows the amperage on the pellet mill motor over the production run. The same challenges were faced with the pellet mill plugging as conditioning temperature was increased. The amperage surges noted on the chart correspond with the pellet mill beginning to plug and the conditioning temperature exceeding 150° F.

As observed in the pilot pelleting experiment, the addition of glycerol in the diet improved pellet quality slightly. The standard and modified PDI for the control diet was 84 and 82%, respectively, compared to 87 and 83% obtained from the diet with glycerol. The same challenges were faced with the pellet mill plugging as conditioning temperature was increased. Figure 2. shows the amperage on the pellet mill motor over the production run. The amperage surges noted on the chart correspond with the pellet mill beginning to plug and the conditioning temperature exceeding 150° F.

Discussion

Collectively, these experiments address practical issues associated with handling and feeding glycerol, and provide a basis for comparing glycerol with other feed ingredients. As discovered in this study, the inclusion of glycerol in diets has advantages relative to feed quality. Pellet quality increased with the use of glycerol; however, conditioning temperature appears to be restricted in diets containing crude glycerol.

Moisture content of the mash is thought to be of strict importance in the prevalence of the mill plugging. As steam conditioning temperature increased, the amount of moisture introduced into the system also increased causing difficulty passing through the pellet mill. Because the viscosity of glycerol changes with steam addition, when reaching higher temperatures and requiring more steam the mash became too wet and plugged the die. Previously stated, the addition of water would improve handling characteristics of crude glycerol; however, evidently with pelleting this is not the case. Additionally, another factor that should be considered when dealing with diets containing glycerol is die retention time. According to Behnke (1994), individual ingredients require a specific amount of time in the die to bind together and form an acceptable pellet. In commercial mills the die working area is much larger, allowing a longer retention times, which mainly used in larger scale facilities because an increased die area allows for greater production capabilities. However, if the die area is too large, the horsepower may be inadequate to provide enough starting torque or prevent mill plugging. At Don's Farm Supply we experienced trouble with the mill plugging during start up. Noted in the graph, there were several surges during starting up of the system. Once the mill ran for some time at a lower production rate we were able to essentially "warm-up" the mill and produced a 3% glycerol broiler diet for

about 20 minutes at 41 tons per hour until our batch ran out, which are ideal operating conditions at the facility.

Implications

Pellet quality was shown to improve with the addition of glycerol in all inclusion levels. Appearance of pellets was acceptable for diets containing 3% glycerol. Diets containing glycerol and steam conditioned higher than 150° F should be closely monitored to refrain from mill plugging. Energy consumption was not significantly impacted. Based off of these findings specific to the equipment used, crude glycerol is a viable option for energy substitution but suggested in levels of 3% or less. Steam conditioning should be kept to a minimum due to potential pellet mill complications.

Figure 4 Pellet mill amperage over time during production with 3% glycerol

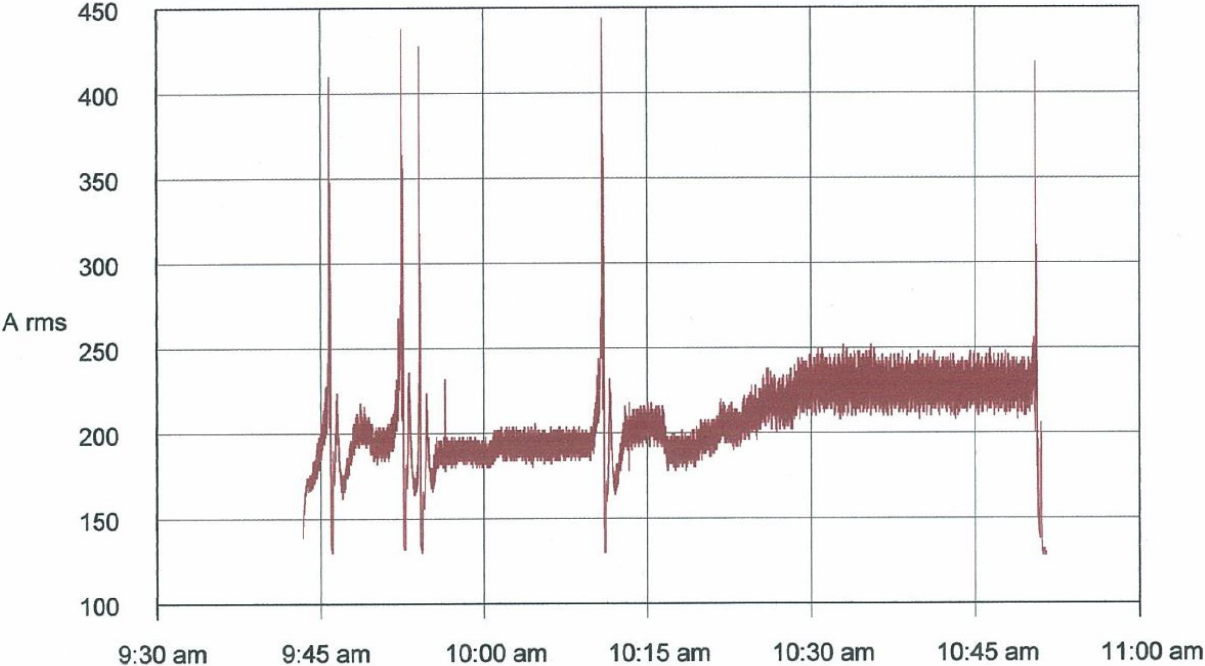


Table 2.3 Composition of experimental diets, Exp. 1

Ingredient	Added Glycerol, %			
	Control	3%	6%	9%
Corn	69.03	66.03	63.03	60.03
Soybean Meal, 48.5% CP	24.64	24.64	24.64	24.64
Soybean Oil	3.29	3.29	3.29	3.29
Glycerol	0	3	6	9
Limestone	1.39	1.39	1.39	1.39
Monocalcium Phosphate, 21% P	1.03	1.03	1.03	1.03
Salt	0.37	0.37	0.37	0.37
Poultry Vitamin/Trace Premix NB 3000	0.25	0.25	0.25	0.25
Total	100	100	100	100

Table 2.4 The effects of crude glycerol on pellet mill performance and feed quality, Exp. 1

Treatment		Response Criteria			
Glycerol, %	Cond. Temp. °F	KWH/Ton	Standard PDI ¹ , %	Modified PDI ¹ , %	Δ Temp. ² , °F
0		6.63	80	74	4.08
3		6.26	88	85	3.47
6		6.99	94	92	3.62
9		6.78	95	94	4.43
	149	6.74	87	82	7.11
	171	6.68	88	86	4.18
	190	6.59	92	91	0.41
Source of variation		Probability			
Glycerol		0.2995	<.0001	<.0001	0.9451
Linear		0.3389	<.0001	<.0001	0.7947
Quadratic		0.1087	0.2311	0.1926	0.8088
Conditioning Temp.		0.9094	0.0028	0.0013	0.0019
Linear		0.6701	0.0007	0.0003	0.0006
Quadratic		0.9527	0.5377	0.4738	0.7356

Chapter 3 - Utilizing DDGS in Animal Diets: Pellet quality and Pellet Mill Performance

Introduction

Distillers dried grains with solubles (DDGS) have been fed to animals for decades. The protein-rich, cost effective ingredient is commonly included in poultry diets as a corn or soybean meal replacement. Traditionally, most integrated commercial facilities producing feed for poultry, manufacture pelleted diets because of the improved performance and growth efficiency that offsets the cost of pelleting. However, few studies have evaluated the use of DDGS in pelleted diets on pellet quality and energy consumption. Fahrenholz et al., 2008 reported the ability to pellet a corn-soy based diet containing 40% DDGS while maintaining comparable energy consumption levels to the control diet containing no DDGS. However, pellet quality, throughput and bulk density was negatively affected by the addition of DDGS.

Steam quality and conditioning temperature are two key criteria associated with pellet quality. Research supports the idea that as conditioning temperature increases steam penetration per particle improves, which can potentially lead to improved gelatinization and better binding and pellet formation through the die. Tumuluru, 2009 reported that increasing conditioning temperature improves pellet quality in diets containing wheat distillers dried grains with solubles. However, diets containing corn DDGS require different operating factors in order to reach optimum pelleting capabilities. Therefore, this study aimed at adjusting the steam conditioning temperature to determine if additional steam would improve throughput, energy consumption and pellet quality.

Materials and Methods

This experiment was conducted at the Feed Processing Research Center in the Department of Grain Science at Kansas State University and reviewed the effects of including varying levels of DDGS and increasing conditioning temperature on pellet mill efficiencies and pellet quality. Corn DDGS were obtained from a local supplier in Clay Center, KS. A 30 HP California Pellet Mill (Crawfordsville, IN) 1000 series “Master HD” model pellet mill equipped with a 3.97 mm (5/32”) x 31.75 mm (1 ¼”) (hole diameter vs. effective die thickness) pellet die was used to pellet all diets. A Bliss Industries (Ponca City, OK) 30.5 cm (12”) by 121.9 cm (48”) steam conditioner was used to condition feed prior to pelleting. All treatments were conditioned with the same retention time, which was set using a variable frequency drive (VFD). The mash feeder screw was also set to a constant RPM for all treatments using a VFD. Poultry diets were formulated to contain 0, 15 and 30% DDGS and steam conditioned to 140, 160 and 180° F by adjusting the steam flow rate. Pellets were cooled using a double-pass perforated deck cooler (Wenger Manufacturing, Sabetha, KS). All experimental runs were performed using a warm pellet die.

DDGS were substituted for only corn in the diet and no attention was given to the overall nutritional balance of the diet to eliminate any interference associated with variation of ingredients on pellet quality or pellet mill performance. The results from this study indicated positive results for improved pellet quality and energy consumption in poultry diets containing DDGS by increasing the steam conditioning temperature.

Data Collection

Pellet mill production data was collected on all diets. Each diet run was replicated in triplicate by manufacturing a new batch of feed each time. Pellet mill electrical consumption

(kWh/ton), production rate (t/hr), hot pellet temperature (°F) and pellet durability (%) were measured. Electrical energy data was collected using an Amprobe (Miami, FL) DM-II (Pro) Data Logger Recorder. This data was used along with the determined production rate to calculate and report pellet mill electrical consumption in kilowatt-hours per ton (kWh/ton). Production rate was measured directly at the pellet mill by collecting pellets at the discharge for 15 seconds, weighing and calculating production in kilograms per hour. Samples were taken consecutively three times at equal intervals and an average value was determined and used as the production rate for the run. Conditioning temperature was measured through a stiff thermocouple placed in the steam of the conditioned mash as it moved from the conditioner to the pellet die and was adjusted by controlling the steam flow rate. To measure the hot pellet temperature, pellets were collected in a circular motion in an insulated foam pail, and the temperature was taken using a stiff thermocouple after the temperature equilibrated. Pellet quality was measured and reported as the pellet durability index (PDI) using the tumbling box procedure ASAE S269.4 (ASAE, 1991). Two standard and two modified (inclusion of five 12.7 mm (1/2”) hex nuts) PDI tests were conducted for each production run and an average value for each was reported.

Statistical Analysis

This study was conducted using a 3 x 3 factorial randomized complete block design (RCBD) consisting of three treatments containing 0, 15, and 30% DDGS steam conditioned to 140, 160, and 180° F. Statistical analysis was performed using the general linear model (GLM) of SAS (v. 9.2). Analyses were completed for standard PDI, modified PDI, energy usage, and production rate. Contrasts for linear and quadratic polynomial effects of glycerol based on diet and conditioning temperature were also included in the analysis.

Results

Results of this study are presented in Table 3.2. There was a significant interaction between conditioning temperature and level of DDGS in the diet on energy consumption ($P < .01$) and production rate ($P < .056$). Increasing DDGS inclusion had a significant ($P < .001$) effect on production rate. Pellet durability showed significant improvement in both standard ($P < .006$) and modified ($P < .005$) values. Appearance was acceptable in all diets but greatly improved as temperatures reached 180° F. Energy consumption was comparable across all diets.

Discussion

This experiment was designed to determine if a relationship exists between conditioning temperature and DDGS inclusion level in the diet. In diets containing DDGS, pellet quality was significantly improved as conditioning temperature increased. Treatments formulated to contain 30% DDGS conditioned to 140° F had the poorest quality pellets and required the most energy consumption. As expected, energy consumption significantly improved as DDGS inclusion level and conditioning temperature increased; however, production rate decreased with the addition of DDGS. The decreased production rate can be described from the decrease in bulk density as DDGS were added to the diet.

As conditioning temperature increases, more steam and moisture is introduced into the system allow for more lubrication through the die and decreasing overall throughput and energy consumption. Additionally, the increased temperature improves available steam penetration per particle, which can potentially improve starch gelatinization, resulting in better pellet binding through the die and overall improved pellet quality. Finally, mash bulk density greatly affects

overall production rate of the material. For this experiment, a volumetric feeding device (VFD) was used set the mash feeder screw to a constant RPM to control production rate or energy consumption. Since the screw conveyor is a volumetric metering device the mass discharge rate dependent on the bulk density of the mash. As DDGS are added to the diet, the bulk density decreases and the feeder screw continues to move at the fixed speed resulting in a decreased production rate.

Implications

Pelleting was show to greatly improve as conditioning temperature increased with increasing DDGS inclusion levels. Production rate decreased due to the decrease in mash bulk density as DDGS inclusion level increased in the diet. Energy consumption improved as conditioning temperature increased because of the additional moisture serving as a lubricant in the die.

Table 3.5 Composition of experimental diets

Ingredient	Control	DDGS Level, %	
		15	30
Corn	74.44	59.44	44.44
DDGS		15.00	30.00
Soybean Meal	20.4	20.4	20.4
Monocalcium Phosphate	0.51	0.51	0.51
Limestone	0.58	0.58	0.58
Salt	0.33	0.33	0.33
DL-Methionine	0.31	0.31	0.31
Lysine HCl	0.18	0.18	0.18
Poultry Premix	0.25	0.25	0.25
Soybean oil	3.00	3.00	3.00
TOTAL	100.00	100.00	100.00

Table 3.6 The effects of DDGS on pellet mill performance and pellet quality

DDGS, %	Cond. Temp. °F	KWH/Ton	Standard PDI¹, %	Modified PDI¹, %	lbs/Hr
0	140	9.25	64	52	2818
	160	7.35	71	62	2802.67
	180	6.31	82	75	2837.33
15	140	8.39	56	43	2748.33
	160	6.79	66	56	2788
	180	6.05	79	74	2798.67
30	140	7.95	45	29	2634.67
	160	6.28	61	48	2689.33
	180	6	78	71	2695.33
Source of variation		Probability			
DDGS		<.0001	<.0001	<.0001	<.0001
Linear		<.0001	<.0001	<.0001	<.0001
Conditioning Temp.		<.0001	<.0001	<.0001	0.0005
Linear		<.0001	<.0001	<.0001	0.0001

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