UDK 636.085.2:577.161.3:622.788
Original research paper

EFFECT OF PELLETING AND EXPANDING PROCESSES ON STABILITY OF VITAMIN E IN ANIMAL FEEDS

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ABSTRACT: The stability of vitamin E (DL-alpha-tocopherol-acetate) in pelleted feed for broilers and expanding feed for pigs during storage in controlled, but extreme conditions (at temperature 60 ºC and relative humidity 80%) was studied over a period of three months. Determination of vitamin content was performed by Liquid Chromatography (HPLC). During storage for three months, the concentrations of vitamin E in untreated feed samples for broilers and pigs decreased to 62% and 74% of their initial values, respectively. In the samples of pelleted feed for broilers and expanding feed for pigs, the concentrations of vitamin E decreased to 46% and 53% of their initial values, respectively. Increase of vitamin E content, between pelleting and expanding feed samples, was not statistically significant, but shows that the pelleting process has a greater impact on the degradation of vitamin E than expanding process. Losses, in general, were in the range found in other studies.

Key words: *pelleting, expanding, stability, vitamin E, HPLC*

INTRODUCTION

Feed supplements are substances, microorganisms or preparations, together with finished products of animal feed and premixes, which are intentionally added to animal feed or drinking water for animals with the aim of conducting one or multiple functions. They are also used for more efficient tehnology production process in preparation of the mixture, the improvement of the characteristics of products of animal origin, as well as increasing the efficiency of environmental protection. Nutritional supplements in feed, whose specific effects on the animal organism are dosedependent, are the chemicals necessary for life and growth of organisms.

Many ingredients in food naturally contain certain nutritional values (they are rich in specific vitamins, minerals…) (Coelho, 2002).

Vitamins are essential for growth, health, reproduction and survival. They are involved in over 30 metabolic reactions in cellular metabolism and critical to the efficiency of the Krebs/Citric Acid cycle (Marks, 1979). Vitamins are present in most common feedstuffs in minimal amounts and because they are necessary for normal metabolism, cause a specific deficiency disease if absent from the diet. Generally, vitamins are quite sensitive to their physical and chemical environment (Coelho, 2002).

Vitamin E, as DL–alpha–tocopherol, is an antioxidant by itself and, therefore, if applied directly to feeds, is consumed rapidly. The free phenolic hydroxyl group in this molecule is responsible for the antioxidant activity. When the hydroxy group is protected by formation of an ester, as in tocopheryl acetate, the compound obtained is resistant to oxygen, since it has no double bonds and free hydroxyl groups.

Feed processes tend to improve the distribution of nutrients and the digestibility of carbohydrates (pelleting, extrusion). However, these processes are harmful to labile nutrients, such as vitamins, that can be easily oxidized (Gadient and Fenster, 1994). Conditioning temperatures have increased to reduce microbial contamination and improve pellet quality and/or production rate. From a practical standpoint, vitamin activity may be decreased in finished feeds due to heightened conditioning parameters. Heat, friction, steam (moisture), pressure, and oxidation are some factors that influence vitamin stability during feed processing (Anderson and Sunderland, 2002; Sredanović et al., 2003). Gadient and Fenster (1994) reported that moisture addition influences vitamin activity more than conditioning temperature. Moisture softens the coating of vitamins allowing oxygen and other compounds access, thus creating vitamin destruction by accentuating chemical reactions. Oxidation−reduction reactions are primary concern with vitamin activity.

Pellet conditioners, expanders, and extruders are the three basic types of processing used in the feed industry. Pellet mill conditioners are used to add steam prior to pelleting (Vukmirović et al., 2010; Čolović et al., 2010). The addition of steam increases feed temperature of the mash feed in the range of 85 to 100 ºC for approximately 20 seconds. Extended pellet mill conditioners enable the increased temperature to be subjected to the feed as much as 20 minutes, which is dependent upon the length of the conditioner. The increase in conditioning time allows for increased starch gelatinization, protein denaturation, pathogen reduction, improved pellet quality, and increased production rate (Sredanović et al., 2005). These processes are intended to increase the value of feed ingredients. For example, the digestibility may be increased or the palatability improved (Riaz, 2007). On the other hand, microingredients such as vitamins are more likely to be damaged by the feed manufacturing process. Vitamin bioavailability is affected by the stability of the vitamin and the utilization efficiency (Baker, 1995).

In this study, the stability of formulated commercial form of tocopherol−acetate (vitamin E) was evaluated in pelleted feed for broilers and expanded feed for pigs.

MATERIAL AND METHOD

Samples

Four samples of animal feed were examined:

1. Complete mixture for broilers (untreated) − UTB
2. Pelleted feed for broilers: − PEB

   Pelleting conditions: Complete mixture was conditioned in double−shaft steam conditioner Muyang SLHSJ0.2A (China), until material reached temperature of 80 °C, with direct water addition into feed mash during conditioning. Material moisture content after conditioning process was 15.5%.

   The material was pelleted on a flat die pellet press 14−175, AMANDUS KAHL GmbH & Co. KG (Germany). A die with 6 mm diameter of the openings and with press way of 36 mm was used (diameter ratio 1:6). The pellets were collected at pelleting temperature of 60 °C. The rate of product flow was 18.6 kg/h. After pelleting, pellets were sto-red for 24 hours under room conditions in order to achieve stabile temperature and then were milled by hummer mill with sieve opening of 4 mm.
3. Complete mixture for pigs (untreated) − UTP
4. Expanded feed for pigs: − EXP

   Expanding conditions: Complete mixture was conditioned in double−shaft steam conditioner Muyang SLHSJ0.2A (China), until material reached temperature of 80 °C, with direct water addition into feed mash during conditioning. Material moisture content after conditioning process was 25.50%.

   A single screw annular gap expander (OEE 8, AMANDUS KAHL GmbH&Co. KG, Germany) with a length−to−diameter ratio of 8.5:1.0 and capacity of 100 kg/h was used for obtaining expanded product at 130 ± 1 °C. The speed of passage of material was 15.84 kg/h. The product was stored for 24 hours under room conditions in order to achieve stabile temperature and then was milled by hummer mill with sieve opening of 4 mm.

One kilogram of each feeds for broilers contained minimum 30 mg/kg of vitamin E and each samples of feed for pigs II contained minimum 40 mg/kg of vitamin E. Vitamin E contained in the samples was in the form of coated tocopherol–acetate. Samples were stored in glass bottles (370 ml volume) in the Climate chamber Binder KBF series (E5.2), (Binder GmbH, Tuttlingen, Nemačka), in the dark at the temperature of 60ºC and at the relative humidity of 80%, and analysed at the beginning of the study and after each week, a total of three months (12 weeks).

Reagents

Methanol (HPLC grade), methanol, ethanol (extra pure grade) were purchased from Sigma–Aldrich. The vitamin E standard (DL–alpha Tocopherol–acetate, analytical standard min. 99.9%, Lot. No.: LB87728V) were obtained from Supelco analytical, USA.

Stock solution of vitamin E with the concentration of around 1mg/ml was prepared by dissolving 1 mg in 10 ml of 2–propanol. This stock solution was used to prepare a series of standard solutions for calibration curve.

HPLC–DAD

The HPLC instrument was an Agilent 1200 system equipped with a diode array detector (DAD), Chemstation Software (Agilent Technologies), a binary pump, a vacuum degasser, an auto sampler and Agilent column (Zorbax eclipse plus-C18, 1.8 μm, 2,1x100 mm). Methanol was used as mobile phase at a flow–rate of 0.3–ml/min and a pressure of 230 bars. Five microliters of standards and samples was injected onto the HPLC column. The spectra was recorded at 285 nm. Identification of DL–alpha–tocopherol–acetate was done by comparing the retention times and spectra of DL–alpha–tocopherol–acetate from samples and standards.

Extraction procedure

Vitamin E–acetate extraction and the cleanup procedure were performed according to the method described by Rushing (Rushing et al, 1991) with slight modifications. Around 20 g of each feed type was extracted in 250–ml screw–cap Erlenmayer flasks with 50 ml portions of extraction solvent (methanol/ethanol, 60:40, v/v) using ultrasonic bath (VIMS elektrik, Loznica). After extraction, extract was filtered through 0,45 μm pore size PTFE filter (Plano, Texas, USA) and the filtrate was transferred to an HPLC vial.

RESULTS AND DISCUSSION

Thermal processing (pelleting, roasting, expansion, and extrusion) of animal feeds is common as a way to improve growth rate, efficiency of gain, nutrient digestibility, and to improve feed handling (Fairfield, 2003). Thermal processing has been shown to reduce effectiveness of functional protein ingredients such as enzymes and less stable nutrients such as vitamins (Dozier, 2002).

The results of the determination of vitamin E in investigated samples of animal feed, are presented in Table 1.

Table 1.
The average concentrations of vitamin E (mg/kg) in samples of pelleted feed for broilers and expanded feed for pigs and the fraction of their initial concentrations at the beginning of the study and after 1, 2 and 3 months.

Feed samples	Initial values	1 Month	2 Months	3 Months
UTB	35.2 ± 1.6	32.1 ± 2.3	25.8 ± 1.7	21.8 ± 1.6
PEB	33.5 ± 1.7	25.1 ± 1.9	20.9 ± 2.3	15.4 ± 2.9
UTP	43.5 ± 2.1	39.3 ± 3.0	35.6 ± 2.6	32.2 ± 2.5
EXP	42.7 ± 2.3	35.4 ± 3.7	26.7± 3.1	22.6 ± 1.9

n=6; UTB- complete mixture for broilers (untreated); PEB- pelleted feed for broilers; UTP- complete mixture for pigs (untreated); EXP- expanded feed for pigs.

At the beginning of the study, the concentrations of the tested vitamin E in the initial samples of complete mixture for broilers and complete mixture for pigs were in the amount of prescribed by the manufacturer (minimum 30 mg/kg of vitamin E in feed for broilers and minimum 40 mg/kg of vitamin E in feed for pigs). But, at the beginning of the study, the concentration of vitamin E in pelleted feed for broilers decreased to 95% of its initial values while its average concentration in the sample of expanded feed for pigs decreased to 98%.

This increase of vitamin E content between pelleted and expanded feed samples, was not statistically significant, but shows that the pelleting process has a greater impact on the degradation of vitamin E than expanding process. In pelleting, the most important adverse factors are friction (abrasion), pressure, heat, humidity and conditioning time. Friction and pressure expose more vitamin molecules to chemical destruction. Heat and humidity accelerate most chemical reactions.

In expanding process, the dominant effects are pressure, heat, humidity and redox reactions. Expanding is hydrothermal process utilizing temperatures higher than 90 °C used to produce more hygienic compound feeds. In an expander, more of the starch is gelatinized and this factor maximizes pellet durability and nutrient digestibility. In addition, expanded feed can be pelleted easily and therefore pellet output is higher, but high temperatures, friction and other processing stress factors can cause losses of some vitamins.

Vitamin E is significantly more stable than other vitamins (Broz et al., 1997), but trace metals in premixes and feeds with moisture content in combination, may be the main contributing factor to loss of α-tocopherol–acetate as suggested by Coelho (Coelho, 2002). Trace minerals has a significant impact on vitamin E stability. Free metal ion is the most reactive (metal filings) followed by sulfate, carbonate, oxide and the least reactive form is chelated. Friction is also an important factor because it erodes the coating that protects several vitamins and reduces vitamin crystals to a smaller particle size.

During storage for three months in extreme conditions of Climate chamber Binder (temperature 60 ºC and humidity 80%) average concentrations of vitamin E in untreated feed samples for broilers and pigs decreased to 62% and 74% of their initial values, respectively. In the samples of pelleted feed for broilers and expanded feed for pigs, the concentrations of vitamin E decreased to 46% and 53% of their initial values, respectively. After first month the difference in the stability of vitamin E in the untreated feed samples (UTB and UTP) was biger than in the treated feed samples (pelleted feed for broilers and expanded feed for pigs), but was not significant. Vitamin E showed the adequate stability within 3 months, but at the same time considering extreme storage conditions, in a climate chamber, we can conclude that vitamin E could be stable more than 3 months in the storage under normal, controlled conditions.

The stability of the supplemental DL–tocopherol–acetate in mixed feeds used in this experiment indicates that feeds supplemented with DL–tocopherol–acetate will retain their potency for a considerable period of time. The relatively high stability of DL–tocopherol–acetate observed in these experiments confirms the observations of Young et al. (2011).

CONCLUSIONS

Examined vitamin E was more stable in the untreated feed samples for broilers and pigs. The content of vitamin E decreased to 62% and 74% of their initial values, respectively. In the samples of pelleted feed for broilers and expanded feed for pigs, the concentrations of vitamin E, after 3 months in extreme conditions in climate chamber, decreased to 46% and 53% of their initial values, respectively. Increase of vitamin E content, between pelleted and expanded feed samples, was not statistically significant, but shows that the pelleting process has a greater impact on the degradation of vitamin E than expanding process. Losses in content of vitamin E, in general, were in the range found in other studies.

ACKNOWLEDGMENT

The paper is a part of the research work on the project III–46012 funded by the Ministry of Education, Science and Technological Development of the Republic of Serbia.

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УТИЦАЈ ПЕЛЕТИРАЊА И ЕКСПАНДИРАЊА НА СТАБИЛНОСТ ВИТАМИНА Е У ХРАНИ ЗА ЖИВОТИЊЕ

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Сажетак: Витамини, као биолошки активне компоненте, су осетљиви на физичке и хемијске утицаје. Процеси који се примењују при производњи хране за животиње теже да побољшају апсорпцију храњивих материја и сварљивост угљених хидрата. Ови процеси утичу на стабилност појединих нутритиената, као што су витамини, који се лако оксидују. Неколико фактора утиче на стабилност витамина у процесима пелетирања, експандирања и складиштења, а то су: влажност, температура, време кондиционирања, оксидо–редукционе реакције, притисак, трење и светлост.

У раду је испитана стабилност витамина Е (дл–токоферол–ацетат) у пелетираној храни за бројлере и експандираној храни за прасад, у контролисаним, али екстремним условима релативне влажности (80%) и температуре (60 ºC), током три месеца. Одређивање садржаја витамина извршено је течном хроматографијом.

Током три месеца складиштења, у нетретираним узорцима хране за бројлере, садржај витамина Е (дл–токоферол-ацетат) је опао до 62% од почетне вредности, док је у нетретираним узорцима хране за прасад његов садржај опао до 74% од почетне вредности. У узорцима пелетиране хране за бројлере током три месеца складиштења, садржај витамина Е је опао до 46% од почетне вредности, док је у узорцима експандиране хране за прасад смањен до 53% од почетне вредности.

На основу добијених резултата, запажено је да процеси пелетирања и експандирања немају статистички значајан утицај на стабилност витамина Е у храни за животиње, али да је утицај пелетирања израженији. Резултати испитивања у складу су с истраживањима других аутора.

Кључне речи: *пелетирање, експандирање, стабилност витамина Е, HPLC*

Received: 8 October 2013

Accepted: 15 November 2013