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Study of In-storage Stability Parameters of the Feed Additive by Thermogravimetric Analysis (TGA)

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Abstract

Thermogravimetric analysis of the feed additive with immunostimulating effect in young farm animals, enabling determination of the temperature zones corresponding to removal of moisture with different binding energies, was conducted. The research allowed determining the periods of dehydration and solids transformation during thermal exposure of the feed additive as well as the temperature zones that correspond to the release of moisture with different binding forms and energies. The feasibility to predict the shelf life of the feed additive for young farm animals when it is exposed to labile physicochemical factors of the environment will result in a wider range of recommended packaging materials and higher storage stability of the additive.

Keywords: immunostimulating feed additive, thermogravimetric analysis (TGA), bound moisture.

ITRODUCTION

The priority with respect to development of beef husbandry in Russia until 2020 is to create favourable conditions for developing production of compound feed additives, which will increase the competitiveness of the industry and exclude antibiotics from the diets of farm animals. According to the World Health Organization, wide use of antibiotics has led to a real threat to human biosafety. Moreover, unbalanced diets and technology stress cause disturbance of microbial balance and development of dysbacteriosis in animals.

It is particularly important to use safe and efficient feed additives in the first six months of calves' life, when apart from active growth they also develop ruminal digestion, which in turn determines both their development and resistance to diseases.

To enable more efficient feed additive storage and select relevant temperature profiles, one should know the nature of moisture binding with material and determine the temperature ranges where the product structure changes. This work is based on thermogravimetric analysis, which provides the moisture removal mechanism data and identifies the temperature ranges and the amount of moisture removed from the material [1-12].

MATERIALS AND METHODS

For simultaneous thermal analysis, STA 449 F3 Jupiter apparatus (Figures 1, 2) with an S-type sample holder (DSC/TG) in aluminum crucible with a punctured lid was used (empty aluminum crucible with a punctured lid was used as a reference); the measurements were carried out under Class 5.0 nitrogen (active gas flow rate 50 ml/min, protective gas flow rate 20 ml/min).

Figure 1: Simultaneous thermal analyser, model STA 449 F3 Jupiter

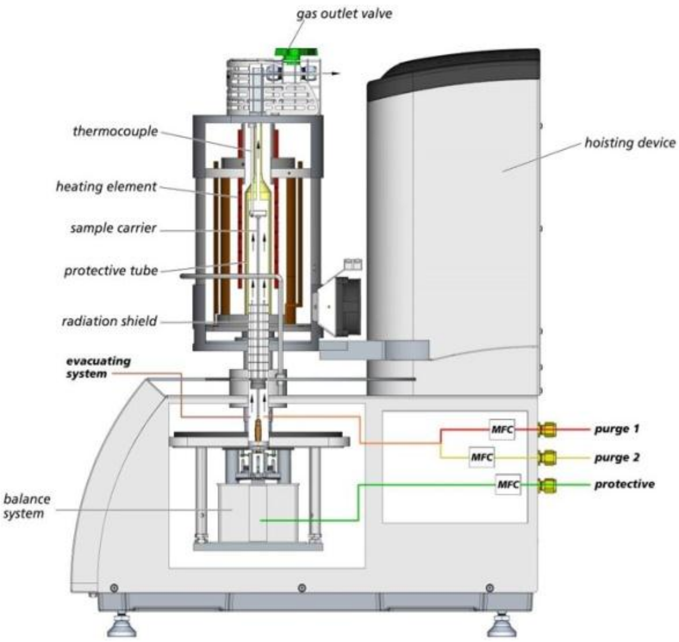

Figure 2: Internal design of simultaneous thermal analyser, model STA 449 F3 Jupiter

The obtained curves were processed using NETZSCH Proteus and Microsoft Excel software.

Thermal analyser's operating principle is based on continuous recording of time- or temperature-dependent mass change of the sample heated according to the selected temperature programme in a gas atmosphere set [1, 2, 8]. Simultaneously, the sample heat release or absorption caused by phase transitions or chemical reactions is recorded [6].

The research was conducted in the following conditions: atmospheric pressure, maximum temperature of 573 K, and the temperature change rate of 10 K/min. The experiments were carried out in aluminum crucibles with a total sample mass of 1.9565 g.

The forms of moisture binding in the product were quantitatively evaluated based on experimental dependences of the material mass change, TG, and the rate of the material mass change, DTG.

RESULTS AND DISCUSSION

Figure 3 depicts the results of thermogravimetric analysis: mass change curve, TG, and mass change rate curve, DTG. As can be seen from the TG curve, monotonous reduction of the material mass was observed with heating, which is associated with moisture loss. In order to obtain the dependence of the degree of mass change (transformation degree), α, on temperature, a portion of the mass change curve, TG, corresponding to dehydration process was used.

The mass of kinetically unequal water molecules in the product was estimated based on the TG curve experimental data by nonisothermal analysis. The portion of the mass change curve, TG, corresponding to the dehydration process (Figure 3) is converted into the material transformation degree, α, as a function of temperature, T. Mass change, Δm_i, corresponding to the amount of water released at the temperature, T_i, (Figure 10) is found on the TG curve for particular temperature values. Transformation degree, α, is calculated as the ratio of mass, Δm_i, to the total amount of water contained in the sample Δm_{max}:

$$\alpha = \frac{\Delta m_i}{\Delta m_{max}}$$

The obtained curve is S-shaped (Figure 4), which reflects a complex nature of the moisture and solids interaction in a material and suggests that different portions of the curve correspond to different dehydration rates.

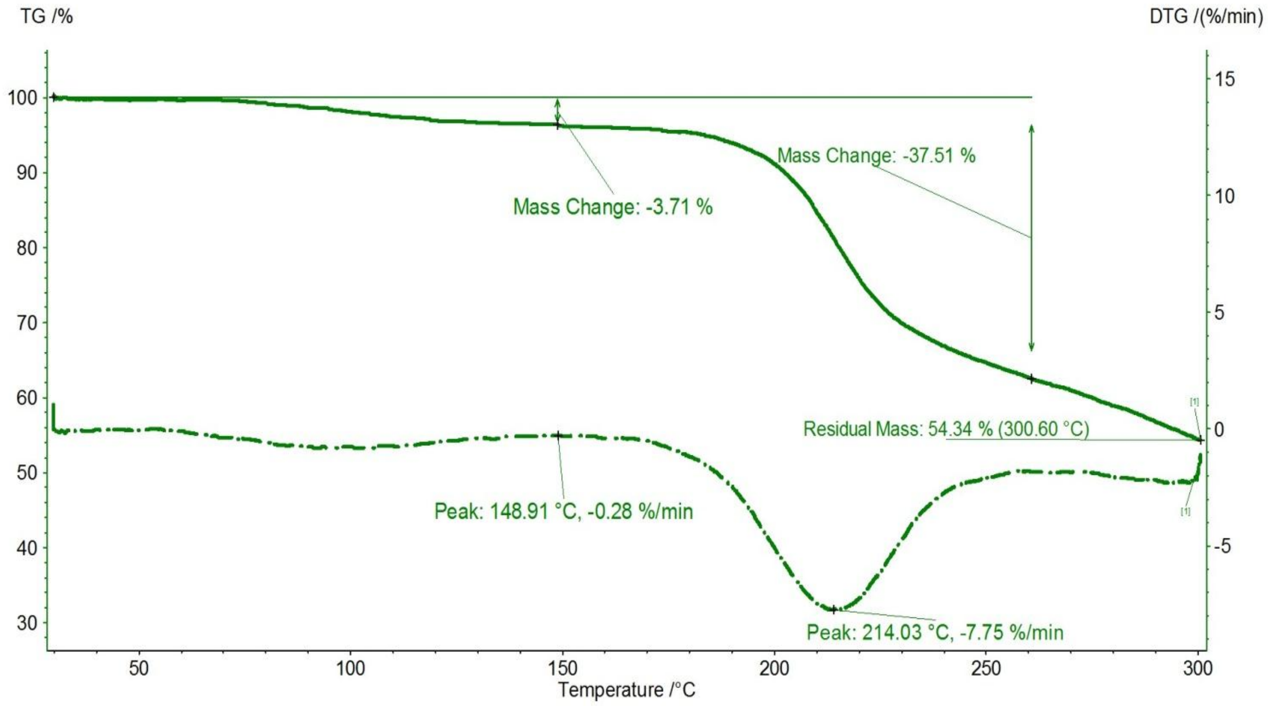

Figure 3: Experimental relationships of the sample mass change, TG, and the rate of the mass change, DTG

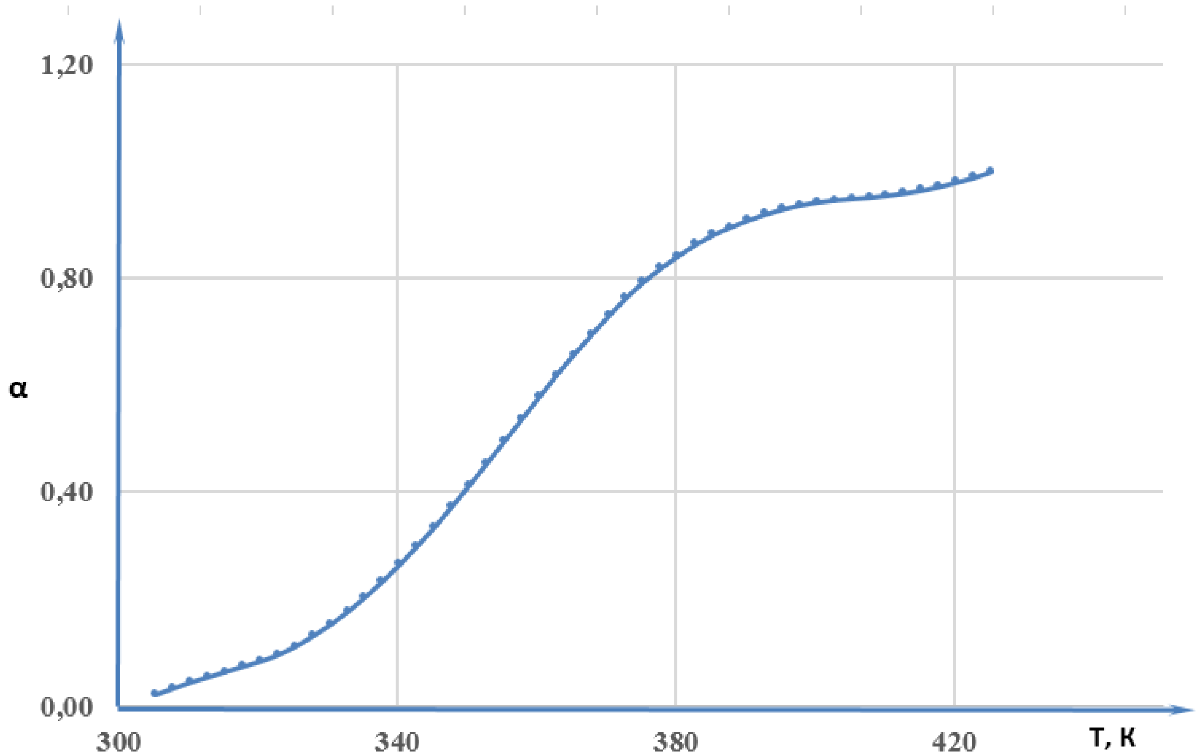

Figure 4: Material transformation degree, α, as a function of temperature, T, of the studied feed additive heated at a temperature rate of 10 K/min

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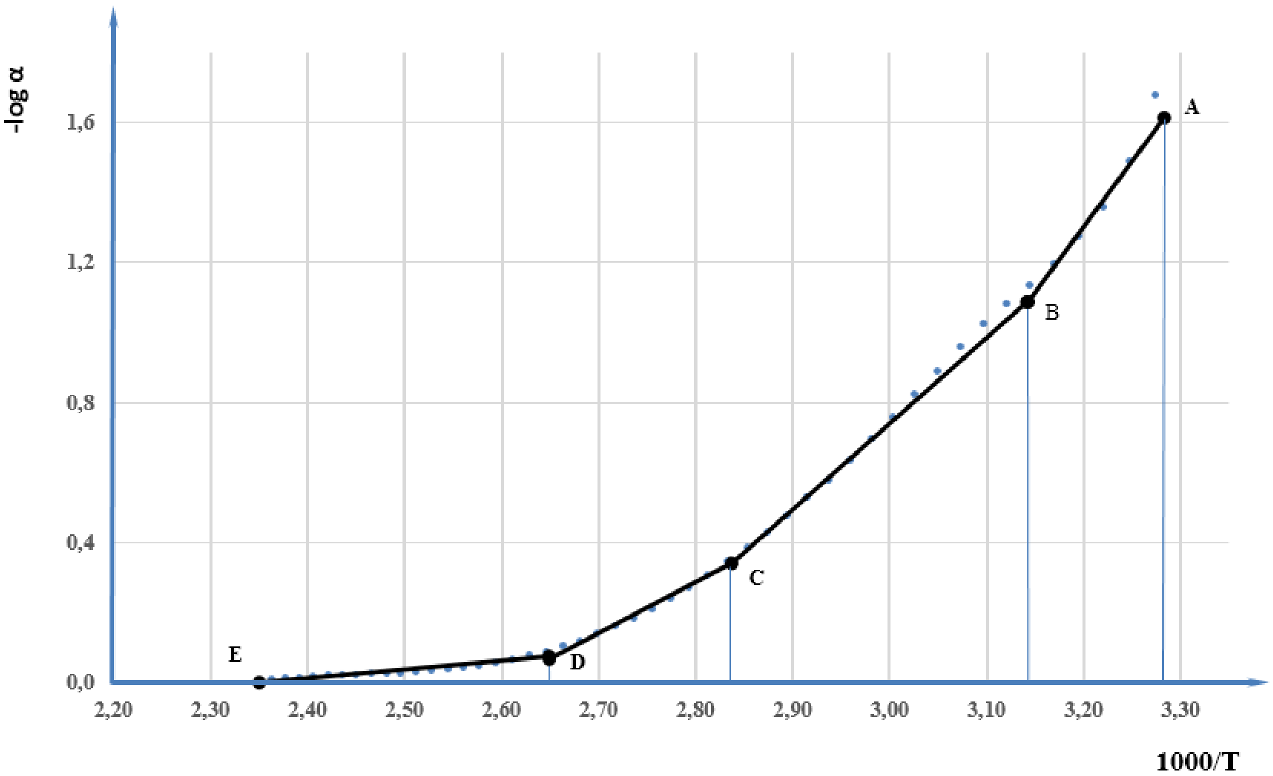

Figure 5: Transformation degree logarithm, (-lgα), as a function of 103/T value while heating at the temperature increase rate of 10 K/min

Material mass (TG curve) reduced with the temperature increase, which is associated with the loss of moisture. The dependence of the temperature change rate is characterized by an endothermic effect in the temperature range of 320...443 K, which corresponds to the maximum product dehydration rate and is accompanied by intensive loss of the material mass.

A curve in (-lg α) - (103/T) coordinates was used to obtain data on the mechanism of moisture removal from the plotted curves and to determine the temperature range and mass percentage of moisture desorbed at approximately the same rate. In Figure 5, four linear portions are visible suggesting stepwise water release from the feed additive. Each of the dehydration steps is characterized by the release of moisture with a particular binding energy.

At the temperature of 295 ... 351 K (portion 1), free water (mechanically and osmotically retained moisture) with low binding energy to product is heated and removed. Released water forms a thin network of water molecule associates bound together with hydrogen bonds. In this case, desorption of capillary water is characterized by lower activation energy values as compared to water released in the second step of the process.

Osmotic moisture of the feed additive is desorbed at the temperatures of 351 ... 419 K (portion 2).

The temperature range of 419 ... 443 K (portion 3) is characterized by the release of adsorbed moisture, and as the temperature approaches the upper limit of the range, internal osmotic and adsorbed moisture is removed from the feed additive. In this case, the substance can partially decompose. As the temperature increases above 443 K, the significant degradation of substances takes place, followed by charring of the products.

CONCLUSION

The analysis of the obtained data made it possible to identify the periods of dehydration and solids transformation in the feed additive with immunostimulating effect for young farm animals when it is exposed to heat and also determine the temperature zones that correspond to the release of moisture with different binding forms and energies. The ability to predict the shelf life of the feed additive for young farm animals allows expanding the range of recommended packaging materials and improving the feed additive storage stability.

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