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STUDIES ON THERMAL INACTIVATION OF EXTRACTED POLYPHENOL FROM DDGS

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ABSTRACT ■ Distillers Dried Grain with soluble (DDGS) is the solid waste of rice based alcohol other industries. It is a nutritionally important source of protein (40%), fat (6%), fiber (7%), minerals (4.5%) and polyphenols (167mg/100g GAE). Again it is nontoxic and does not contains allergen hence may be used as raw material for food product development and extraction of functional component.-During extraction, the phenolic compound may be inactivated during thermal processing. The thermal inactivation kinetics of polyphenol was studied and reported in this paper. Inactivation rate constant and activation energy were calculated. The experimental results were compared with different model equations. Different kinetic data available from this study may be used for prediction of polyphenol inactivation pattern in real food matrix.

Key words: DDGS, degradation kinetics, activation energy, model equation.

1. INTRODUCTION

Distiller's dried grains with solubles (DDGS) are the major by-product in the ethanol and bakery industry. It is rich in protein, polyphenols and high antioxidant activity. But utilisation of DDGS is limited. DDGS produced from cereal grains and generally it is used for animal and poultry feed (Dong et.al 1987). In India, bio ethanol production increased from about 52.8 million of gallons in 2007 to over 225 million of gallons in 2016 (RFA analysis of public and private data sources). The process for making ethanol is dry grind method which is followed by grinding (dry milling), slurrying, cooking, liquefaction, saccharification, fermentation, distillation, and coproduct recovery. In coproduct recovery step, the non-volatile components are known as whole stillage, which is centrifuged and produced liquid thin stillage and solid fraction, distiller grain (DG). The thin stillage is concentrated through evaporation into condensed distiller solubles (DS) also known as syrup. While DS, DG, or their combination known as wet distiller grains with solubles (WDGS). Then WDGS is dried and produced distiller dried grain with solubles (DDGS) (Han & Liu 2010).Consequently this low price DDGS can be used for making several food items, matrix

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of encapsulation bead and can be used as packaging film with antioxidant and polyphenols.

Antioxidant helps to give additional health benefits in human beings. The roles of antioxidants are to giving protection against harmful free radicals, which is the cause of several diseases. Natural antioxidants are like vitamins, carotenoids, flavonoids, and polyphenols. Now a days, increasing demand of consuming antioxidants rich foods and utilisation of antioxidants for various purpose, so it is important bioactive component for market. So it is important to minimise the loss of this antioxidant during processing time to meet the market demand. During processing like – blanching, canning, sterilizing, freezing in food industries, these antioxidants are affected due to thermal degradation, dilution and leaching in to the water. So it is important to taking action to minimise the loss of antioxidants during processing (Nambi et.al 2016).

So, objective of this study is to predict the polyphenols losses during thermal processing. The knowledge of kinetics including reaction order, rate constant and activation energy are applied for this purpose. Kinetic study helps to find out the changes in polyphenols content at different temperature for varied duration.

2. MATERIALS AND METHODOLOGY

2.1. Material

2.1.1. Raw material collection:

Rice DDGS is collected for this study and it was procured from IFB Agro Industry Private Ltd. WB, India. The dried samples were stored in sealed plastic packets at room temperature. Size of the rice DDGS particles are uniform.

2.1.2. Reagents:

Gallic acid, Folin-Ciocalteu reagent, Sodium Carbonate and methanol.

All reagents are procured from the company MERCK, SRL and Spectrochem.

1.2. Experimental procedures(Nambi, Gupta, Kumar,&Sharma 2016)

1.2.1. Extraction of phenolics from DDGS:

The DDGS was dried at 40° C. Approximately 10g sample was mixed with 30ml methanol and stirred for 24 h in the dark and at 35ºC using a shaking incubator. After this, the homogenates in the tube were centrifuged at 12,000 rpm 15min at 20ºC and the supernatant was transferred into an amber volumetric flask and made up to a final volume of 100 ml.

1.2.2. Thermal treatment:

For thermal treatment, 100ml of extracted polyphenol sample was taken in a glass beaker and kept in a water bath at 45°C, 55°C, and 65°C separately for 0 to 30 min. Samples were taken out at 15 min interval. After heating at desired temperature, the samples were stored at freeze for assessment of total phenolic content.

1.2.3. Determination of total phenolic content:

The concentration of total phenolics (TPC) in the methanol extract of DDGS samples was determined using the Folin – Ciocalteau colorimetric method. Briefly, a 1 ml sample extract and 10 mL of 7% sodium carbonate were transferred into a test tube. After about two minutes 1 mlFolin-Ciocalteu's reagent was added and mixed thoroughly. And then make up the volume up to 25ml. The mixture was left to stand for 45 min at room temperature in the dark. The absorbance of the reaction mixture was measured at 765 nm in a spectrophotometer. The calibration curve was performed using Gallic acid as standard and the results were expressed as mg of Gallic acid equivalents 100g of sample (mg GAE/ 100 g of sample).

1.2.4. Decay Kinetics(Van Boekel, 2008)

Degradation kinetics was calculated using the following equation

$- d[c] / dt = k[c]^m$..... [1]

Where C is the quantitative value (concentration), k is the reaction rate constant, and m is the order of the reaction. From equation 1, first order can be derived as follows:

$C = C_{0exp} (- kt)$ .....[2]

Frequently, the logarithmic form is used instead of exponential equation:

$\ln c = \ln c_0 - kt$.....[3]

The nonlinear Eq.2 is thus transformed into the linear Eq. 3.

Temperature dependence of a reaction is described by the Arrhenius equation. Arrhenius law was empirically derived to describe the temperature dependence of simple chemical reactions. It was proven to be very important factors in chemical kinetics. It relates the rate constant *k* of a reaction to absolute temperature T:

$K = A \exp (-E_a/ RT)$ ..... [4]

The linearized form is:

$\ln k = \ln A - Ea / RT$ ..... [5]

in which, A is a so called "pre-exponential factor" (sometimes called the frequency factor), Ea the activation energy, and R and T the gas constant and absolute temperature, respectively. The dimension of A should be the same as that of the rate constant *k;* it therefore does have units of frequency only in the case of a 1st order reaction.

The parameter Q_{10} describes the temperature dependence of a reaction as the factor by which the reaction rate is changed when the temperature is increased by 10°C:

$Q_{10} = k_{T+5} / k_{T-5} = k_{T+10} / k_T$ ..... [6]

Another parameter to describe temperature dependence is Z, which expresses the increase in temperature that would produce an increase in rate by a factor of 10. ~~Z is~~

$Z = 2.303 RT^2 / Ea = 10/ \log Q_{10}$ .... [7]

Like the parameter Q_{10}, Z is temperature dependant which restricts its use.

Also used ~~is~~ the parameter D, especially in thermo-bacteriology. It is the decimal reduction value, the time needed to reduce a concentration by a factor of 10. D is nothing other than an inverse rate constant. For a 1st order reaction:

$D = \ln 10 / k = 2.303/k$.....[8]

3. RESULTS

Total Phenolic Content

Polyphenol quantity was determined at different temperature i.e. 45°C, 55°C and 65°C for different time interval.

The total phenolic contents decreased in rice DDGS during heating (Fig.2). When time and temperature of heating increased, the total phenolic compound decreased.There is also a report that high heat sensitivity of total polyphenol even for a short period of heating (Ismail,Marjan, & Foong, 2004). The loss of

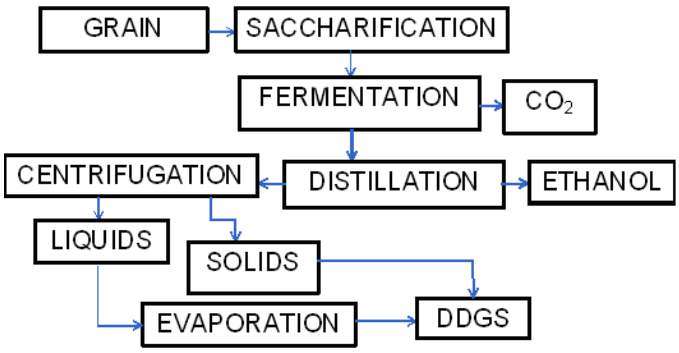

Fig. 1. Flow diagram of production of ethanol and it's by products

phenolic components is due to breakdown of phenolic compound.

The changes in the total phenolic component were plotted against heating time for different temperature (Fig.2).

Polyphenol concentration gradually decreased with time and it is highly dependent on temperature.

45°C, 55°C and 65°C is 0.0047, 0.0057 and 0.006 respectively. At higher temperature molecules are highly energetic and easily take part in reaction.

Now *ln*K at different temperature was plotted against 1/T (Fig.6)

The Fig. 6 showed that the results show a straight line with negative slope. Activation

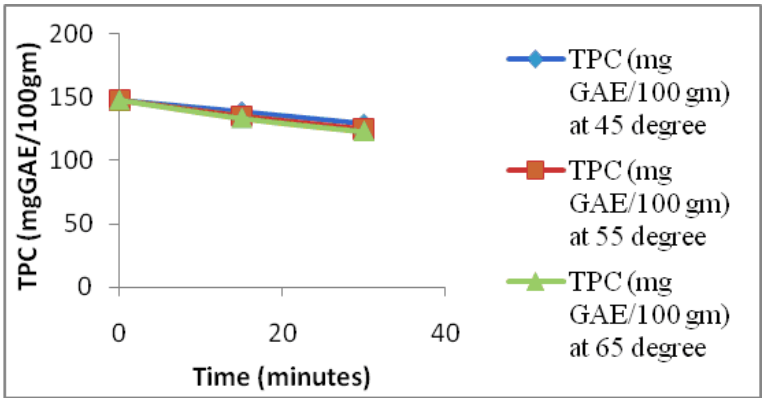

Fig. 2. Changes in total phenolic content at different temperature

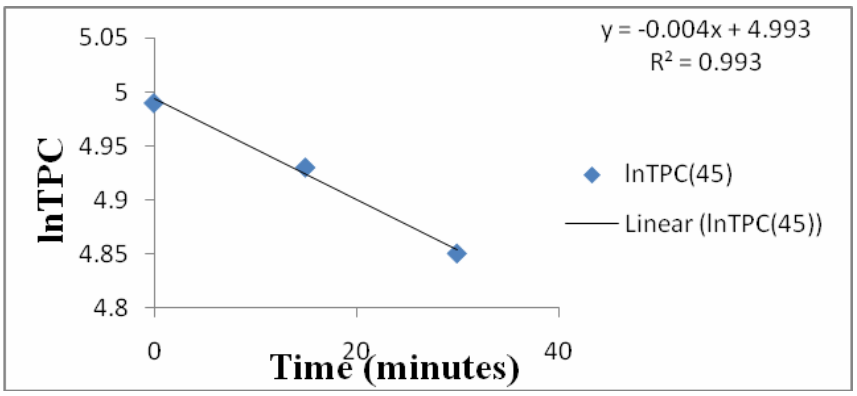

Fig. 3. Determination of reaction rate constant at 45°C

Kinetic model was fitted with the experimental data and the best fit results are given. First order kinetic model was found to be suitable.

Determination of reaction rate constant

It is shown in Fig. 3, 4, and 5 that *ln*TPC*vs* time at different temperature. Fig. 3, 4 and 5 showed that reaction rate constant (K) increases with temperature. The K value of

energy calculated from the slope of this line and its value is 10.184KJ/mol.k.

Decimal reduction value (D) of total phenolic for DDGS decreased with increase in heating temperature. The D valuesindicated that time required to concentration of polyphenolic compound in one log cycle. D values at different temperature are given in table 1.

D values are dependent on temperature.

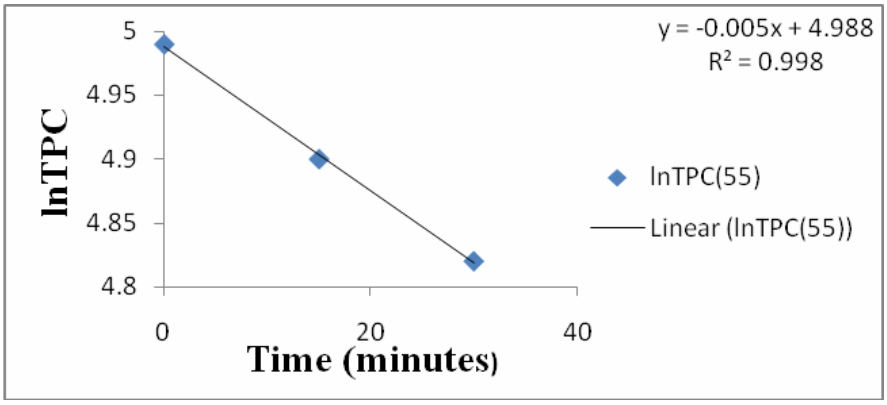

Fig. 4. Determination of reaction rate constant at 55°C

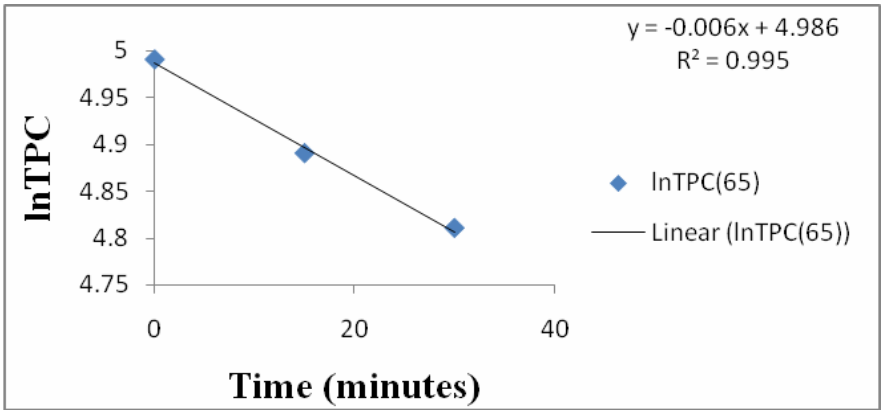

Fig. 5. Determination of reaction rate constant at 65°C

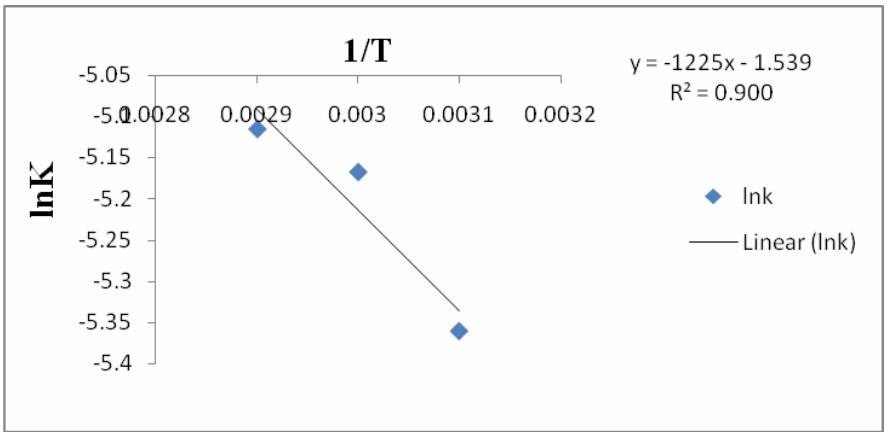

Fig. 6. Determination of activation energy

Temperature dependency of D value is indicated by Z value. Z value is defined as temperatures require reducing one log cycle D value. Hence slope of *ln*D *vs* T (temperature) indicates the Z value (Figure. 7). The Z value is 56.78.

Experimental data revealed that Q10 value is more than 1, i.e. 1.5, which indicates that

Table 1: D values at different temperature

Temperature	D values
45°C	490
55°C	404.03
65°C	383.83

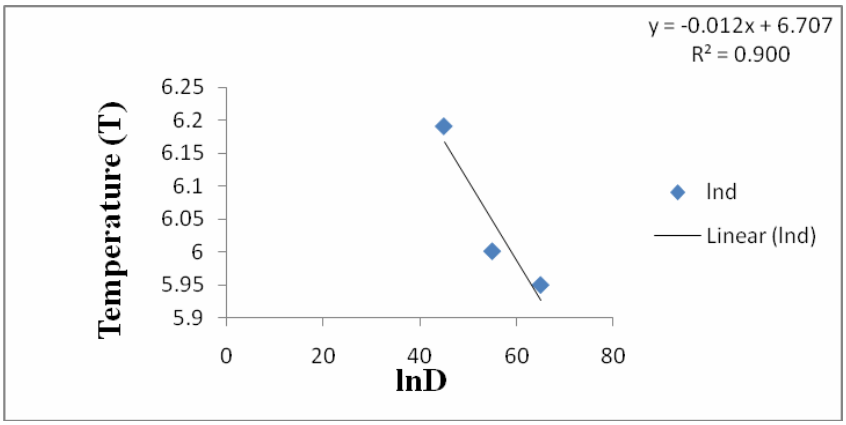

Fig. 7. lnDvs T (indicate the Z value)

there is a positive effect of temperature on degradation of TPC.

4. DISCUSSIONS

It can be concluded that total phenolic content strongly affected by temperature and thermal processing time. Kinetic model revealed the change of phenolic compound effectively. The D value, z, Q_{10} value, and activation energy express the interrelationship of different parameters. These findings would be useful in designing thermal processes of DDGS containing foods.

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