

## Moisture Sorption Hysteresis in *Kalakand*- At Different Temperatures

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### ABSTRACT

The moisture sorption hysteresis is the difference in the equilibrium moisture content between the adsorption and desorption curves. In present the study the moisture sorption hysteresis of *kalakand* prepared from standardized milk were investigated by using static gravimetric method, at 15, 25, 35<sup>o</sup>C, over the range of water activity ( $a_w$ ) 0.11 to 0.97. The hysteresis effect exhibited by *kalakand* shows that the adsorption and desorption isotherms were distinctly apart from each other. The hysteresis loop was classified as Type - C according to Everett and Whitton classification. The distribution of hysteresis loop relative to water activity showed a marked change at various water activities. The hysteresis loop was evaluated in terms of relative hysteresis units. The effect of increasing the isotherm temperature was found decreasing the total hysteresis from 1.72 units at 15<sup>o</sup> C to 1.48 at 25<sup>o</sup> C and 1.13 at 35<sup>o</sup> C. It also resulted in limiting the span of the loop along the isotherms.

**Key words:** *Kalakand*, Water activity, Sorption isotherm, Hysteresis, Hysteresis loop.

### INTRODUCTION

*Kalakand* is important traditional milk based sweet popular all over the India. It is prepared by heating the milk up to boiling and addition of citric acid onset of boiling, with the incorporation of sugar at last stage during manufacturing<sup>9</sup>. *Kalakand* has slightly cooked flavor, brown color, greasy or moist body and grainy texture<sup>1</sup>.

Water is the main component of many food products and the storage stability of products depends upon the interaction of water with other components present in the product.

In the case of dairy products, the storage stability and keeping quality are closely related to the mode of water binding to the other constituents (*viz.* Proteins and carbohydrates). The status of water in product plays a critical role in the preservation of biological materials in both the raw and processed states. The simplest way of expressing such a status is the concept of water activity. It is generally accepted that the water activity is more closely related to the microbial, chemical and physical properties of food products than its total moisture content.

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An important aspect of  $a_w$  concept is the moisture sorption isotherm (MSI) which describe the relationship between equilibrium moisture content (EMC) and water activity ( $a_w$ ) of the food, at constant temperatures and pressures condition<sup>8</sup>. Such relationships help food scientist and technologist to understand water sorption properties of food, particularly when there is a need for selection of ingredient and suitable packaging material and predicting stability and moisture changes during storage<sup>2</sup>.

The difference in the equilibrium moisture content between the adsorption and desorption curve is called hysteresis<sup>7</sup>. In food preservation and storage technology the microbiological stability of stored food items depends more on its equilibrium water activity than on its total moisture content. The hysteresis moisture content appears to be a more important factor than water activity with respect to stability or non-stability of non-microbial quality attributes of food. Hysteresis can be used as an index of total quality as suggested by Wolf *et al*<sup>10</sup>. The increased hysteresis associated with increased moisture content is indicative of reduced quality and while reduced hysteresis and reduced moisture content is indicative of improved non microbial quality attributes like, taste, color and rehydratability of stored products<sup>5</sup>.

Sorption isotherms of several dairy products including *khoa*, *peda*, whey protein, milk powder, cheese and *basundi mix* have been established, but the literature shows no work on moisture sorption hysteresis in *Kalakand*. Therefore the objective of this present investigation was to characterize the moisture sorption hysteresis in *Kalakand*.

## MATERIALS AND METHODS

### Preparation of sample

“*Kalakand*” was prepared using standardized milk (6% fat and 9% SNF) obtained from the Vidya Dairy, Anand, Gujarat, India. The samples of *Kalakand* required for the study was prepared by using a standardized method given by Suresh and Jha (1994). The composition of *Kalakand* was 21.12 % moisture, 21.71 % fat, 15.72 % protein, 17.29 % lactose, 21.52 % sucrose and 2.63 % ash.

### Determination of equilibrium moisture content

The standard sorption apparatus and static gravimetric method recommended by COST 90 project<sup>10</sup> with some modifications were used in determining the sorption equilibrium of *Kalakand*. The sorption apparatus consisted of a wide mouth glass bottle (200 ml) with vapor-tight lid used as sorbostat. Inside each sorbostat there was a support for weighing beaker (25 ml) and the weighing beaker (10 ml) in which the sample material was exposed to the humid atmosphere in the container maintained by the saturated salt slurries. The glass beads were placed in the large beaker (25 ml) to avoid the glass beakers from tilting during handling and weighting.

Seven reagent grade saturated salt solutions in water activity range of 0.11- 0.97 as recommended by Greenspan, 1977 viz. lithium chloride ( $\text{LiCl}\cdot\text{H}_2\text{O}$ ), magnesium chloride ( $\text{MgCl}_2\cdot 6\text{H}_2\text{O}$ ), magnesium nitrate ( $\text{Mg}(\text{NO}_3)_2\cdot 6\text{H}_2\text{O}$ ), sodium chloride ( $\text{NaCl}$ ), potassium chloride ( $\text{KCl}$ ), potassium nitrate ( $\text{KNO}_3$ ) and potassium sulphate ( $\text{K}_2\text{SO}_4$ ) were used to equilibrate the product sample with respective water activity ( $a_w$ ). The sorption apparatus were allowed to equilibrate at respective temperatures for four days before samples were placed into them<sup>6</sup>. For adsorption isotherm, the samples were completely dehydrated by placing them over phosphorous pentoxide ( $\text{P}_2\text{O}_5$ ) for 20 days. Phosphorous pentoxide gives a relative humidity environment close to 0 % and equilibrating the sample with it ensures the complete adsorption isotherm. Weighed quantity of dried sample (Approx.  $1\text{ g} \pm 0.5\text{ g}$ ) was taken into tarred sample beakers, which was then transferred to the sorption apparatus, containing saturated salt slurries. The experiment was conducted in duplicates for best results. The sorption apparatus containing samples were kept at temperatures of 15, 25 and 35<sup>0</sup> C.

The samples for desorption studies were hydrated in a desiccators over distilled water at room temperature to approximately 45 g/100g (d.b.) moisture content to obtain the

complete desorption isotherm. To prevent mold growth, potassium sorbate dissolved the small quantity of water was added @ 0.5 g / 100g of product to “*Kalakand*” sample. The samples were weighed at intervals of 3 days and equilibrium was judged when the difference between three consecutive sample weighing was less than 0.001g. Sample weight in the beaker was recorded with a digital weighing balance (0.001 g resolution). The equilibrium period ranged from 13 to 15 days. Equilibrium moisture content (EMC) in terms of gm of water/100gm of solids was obtained. The values of EMC were plotted against relative humidity to establish moisture sorption isotherm.

## RESULT AND DISCUSSION

### Evaluation of moisture sorption hysteresis

The equilibrium moisture content values during the adsorption and desorption of *Kalakand* reveals hysteresis effect at all three temperatures. The moisture sorption hysteresis of *Kalakand* prepared from standardized milk is shown in Fig 1, 2 and 3 for 15, 25 and 35<sup>o</sup> C respectively. The hysteresis effect exhibited by *Kalakand* shows that the adsorption and desorption isotherms were distinctly apart from each other. The hysteresis loop was classified as Type - C according to Everett and Whitton classification, which begins about 0.9  $a_w$  (upper closing point of loop) and extends over the rest of the isotherm up to 0.11  $a_w$  (lower closing point loop). The distribution of hysteresis loop relative to water activity showed a marked change at various water activities. There was no hysteresis effect in the monolayer moisture content region. It occurred predominantly between 0.40 and 0.76  $a_w$ . The hysteresis effect became minimum beyond 0.85  $a_w$  and the adsorption and desorption isotherm of *Kalakand* coincides with each other at about 0.95  $a_w$  at 15<sup>o</sup> C, 0.93  $a_w$  at 25<sup>o</sup> C and 0.90  $a_w$  at 35<sup>o</sup> C.

Bell and Labuza<sup>3</sup> reported that foods with high sugar content frequently exhibit the phenomenon of hysteresis and explained that when the water moves out from capillaries of the product, during moisture desorption, the

narrow ends of surface pores trapped and held water internally below the water activity where the water should have been released. During adsorption, the pure water would dissolve solutes, that is lactose, sucrose and salts present in *Kalakand* and dissolution of solutes increased the surface tension resulting in lower water activity at given moisture.

A paired t-test was performed using Microsoft Excel-2007 on mean EMC data of adsorption and desorption obtained through three number replication to compare moisture content sorbed by the samples at different temperatures during the both adsorption and desorption. Paired t-test revealed that the adsorption and desorption differ significantly at all the three temperatures with  $t_{0.05} = 4.37$  at 15<sup>o</sup> C,  $t_{0.05} = 4.86$  at 25<sup>o</sup> C, and  $t_{0.05} = 3.08$  at 35<sup>o</sup> C.

The hysteresis loop was evaluated in terms of relative hysteresis units. For this purpose the adsorption values of EMC were subtracted from the respective desorption values of EMC at a given water activity intervals of 0.1 and the resulting difference was plotted versus the corresponding water activity are shown in Figure 4. The graphical integration of area under this curve gave the hysteresis units. The total hysteresis in *Kalakand* was 1.72 units at 15<sup>o</sup>C. The magnitude of hysteresis was smaller at higher temperatures. Yan *et al.* (2008) attributed this phenomenon to the increased elasticity of capillary walls and greater capability of forming hydrogen bond between protein/carbohydrate and water. In *Kalakand* prepared from standardized milk, the effect of increasing the isotherm temperature was found decreasing the total hysteresis from 1.72 units at 15<sup>o</sup> C to 1.48 at 25<sup>o</sup> C and 1.13 at 35<sup>o</sup> C. It also resulted in limiting the span of the loop along the isotherms.

The hysteresis amplitude for a given water activity is characterized by relative water content difference between desorption and adsorption values. The hysteresis amplitude ratio varied throughout the water activity range of 0.11 to 0.96 at all the temperatures. However, the maximum value

for this ratio in *Kalakand* was 0.228 at 15<sup>0</sup> C. It reduced with increase in temperature to 0.225 at 25<sup>0</sup> C and 0.145 at 35<sup>0</sup> C.

In most foods, the sorption capacity decreases with increasing temperatures because of negative excess heat of sorption<sup>4</sup>. The results obtained on *Kalakand* are in agreement to this. Kapsalis (1981) has described three types of hysteresis loops depending upon food component, viz. protein

sugar and starch. In high protein foods moderate hysteresis beginning at about  $a_w$  0.85 extends over rest of isotherm up to zero water activity and both adsorption and desorption remains sigmoid in shape. In high sugar food hysteresis mainly occurs at low water activity and there is no hysteresis above  $a_w$  0.65. As *Kalakand* contains both protein and sugars in fairly large amount therefore it exhibits a combined effect.

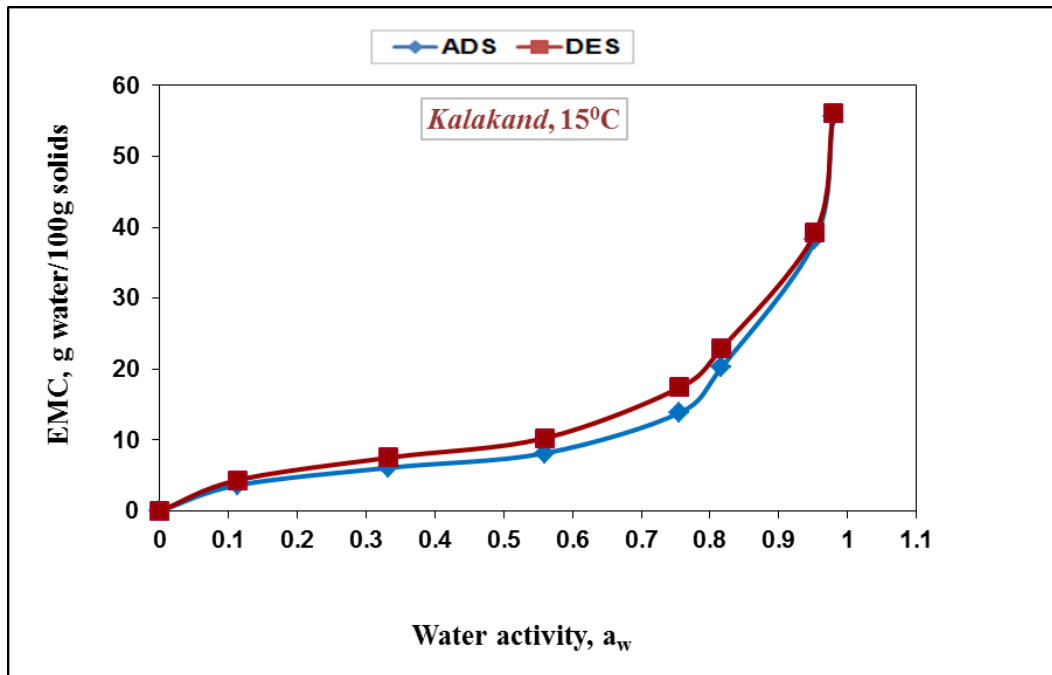


Fig. 1: Moisture sorption hysteresis of *Kalakand* at 15°C

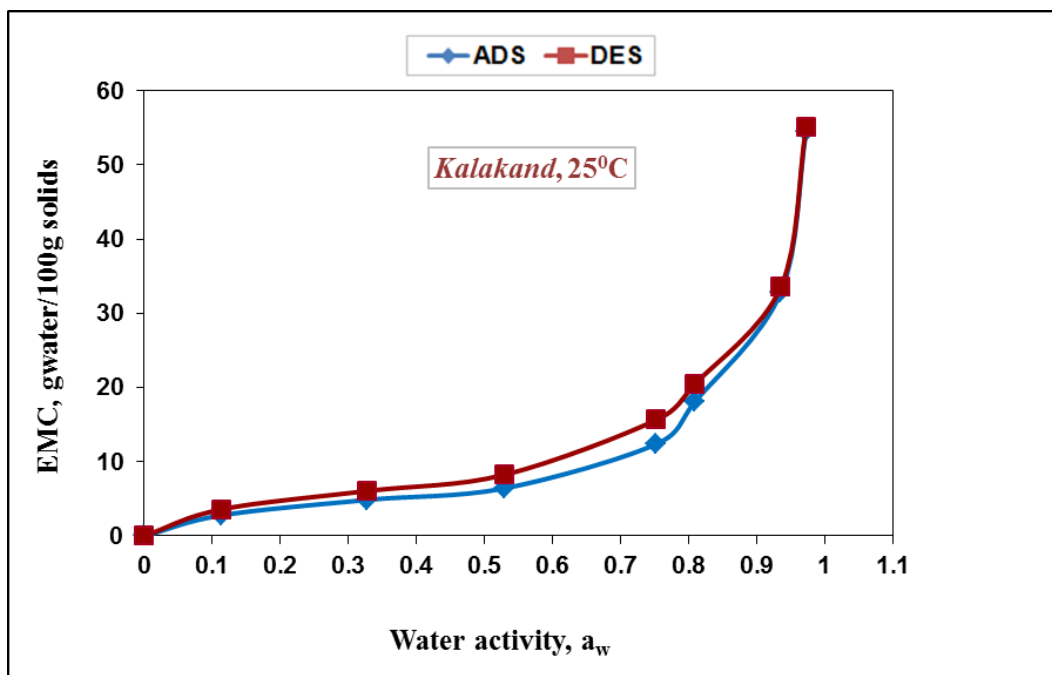


Fig. 2: Moisture sorption hysteresis of *Kalakand* at 25°C

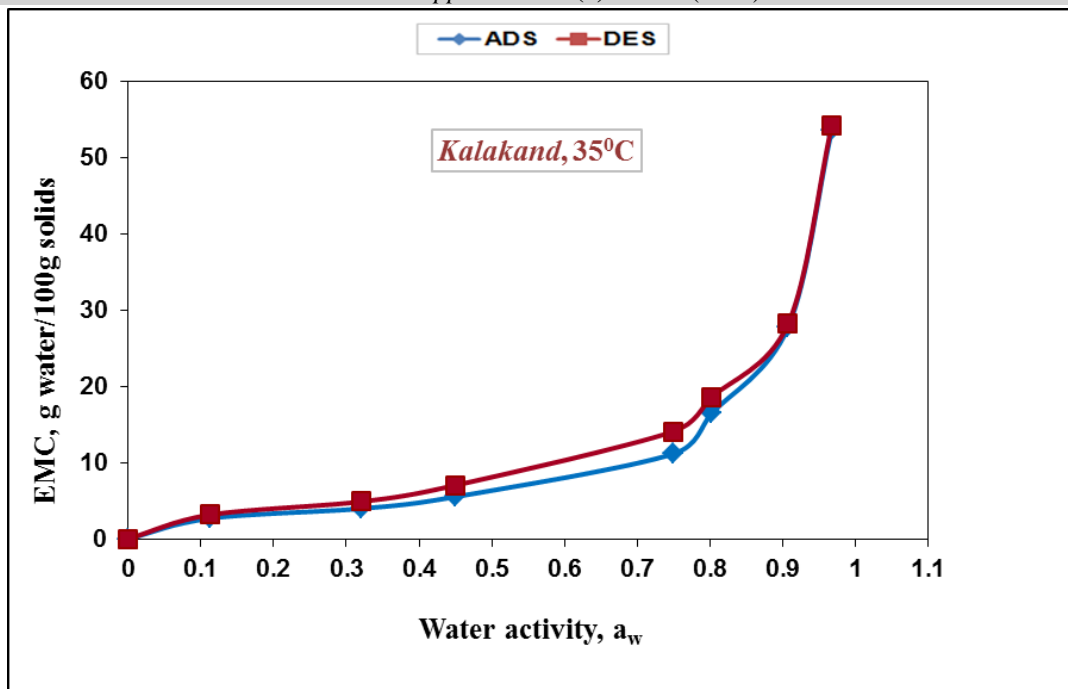


Fig. 3: Moisture sorption hysteresis of Kalakand at 35°C

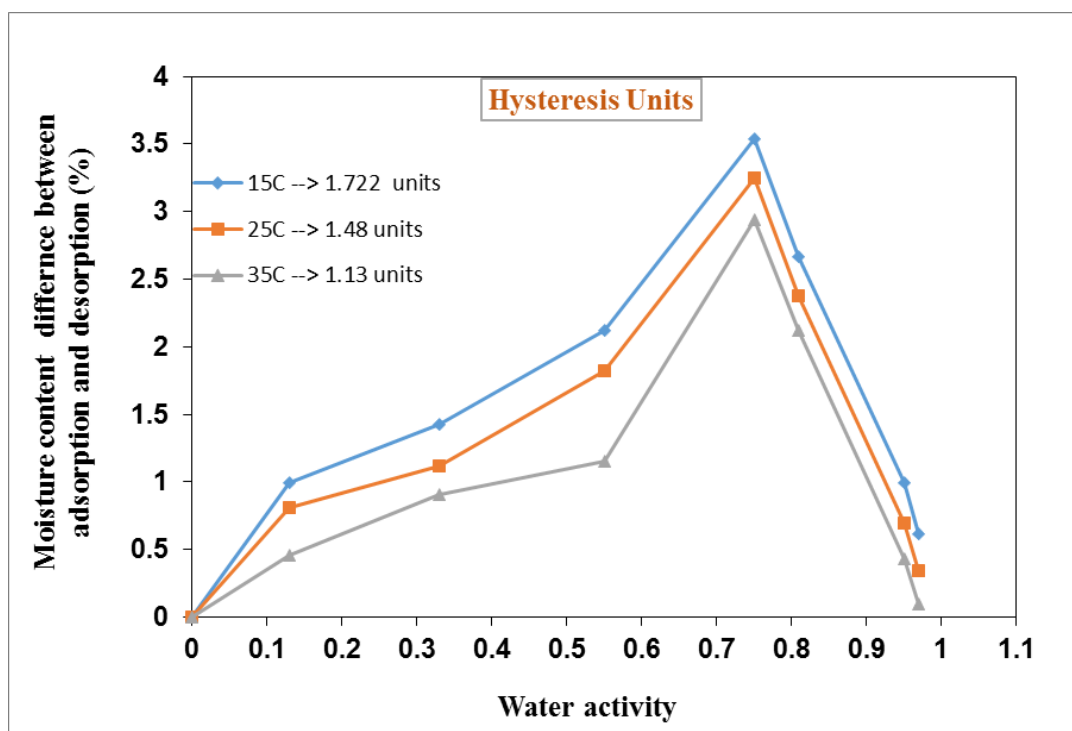


Fig. 4: Effect of temperature on derived hysteresis in Kalakand

**CONCLUSIONS**

The hysteresis loop of *Kalakand* was classified as Type - C according to Everett and Whitton classification. The adsorption and desorption curves in hysteresis loop of *kalakand* were distinctly apart from each other and the hysteresis effect was observed in an entire range of water activities. The hysteresis loop

was evaluated in terms of relative hysteresis units. The effect of increasing the isotherm temperature was found decreasing the total hysteresis from 1.72 units at 15<sup>o</sup> C to 1.48 at 25<sup>o</sup> C and 1.13 at 35<sup>o</sup> C. It also resulted in limiting the span of the loop along the isotherms.

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