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*Research Article*

# Moisture sorption isotherms and heat of sorption of sago starch films

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## Abstract

Moisture sorption isotherms of sago based starch film were determined at 23, 31 and 37$^0$C. A gravimetric static method was used under water activity range of 0.23 to 0.90 for the determination of sorption isotherms that were found to be typical type II sigmoid. The order of fitness of various isotherm models was found to be Halsey> Oswin> GAB > Henderson. The net isosteric heat of sorption, i.e., $q_{st}$ and differential entropy $S_d$ were found to be in the range of 43.12 to 5.01 kJ mol$^{-1}$ and 0.13 to 0.01 kJ mol$^{-1}$ respectively, for the moisture content range of 0.18 to 0.45 g/g dry basis. A fair correlation was found between $q_{st}$ and $S_d$. The values of isokinetic temperature $T_\beta$ and harmonic mean temperature $T_{hm}$ were evaluated to be 311.4 and 303.2 k respectively.

**Keywords:** edible films, Halsey models, Oswin model, GAB model, isosteric, India.

## Introduction

In recent years, a great deal of research work has been dedicated to develop packaging materials for the purpose of protecting food. Edible films are used to control moisture transfer, limit gas transport, retard oil and fat migration, prevent solute or flavour absorption, etc. [1].Many natural polymers, such as polysaccharides and proteins, are preferred to prepare food packaging films to eliminate environmental pollution and hazards caused by petrochemical-based packaging films [2]. Polysaccharides such as starches [3-7)], cellulose derivatives [8-9], plant gums [10], chitosan [11], alginates [12], etc., have been used frequently for preparing packaging films.

As mentioned above, an important role of packaging materials, including edible films, is to reduce the exchange of water between the food and the environment, in particular drying of moist food [13]. In order to predict performance of packaging film, data on physical properties including equilibrium moisture content is needed which may be expressed in moisture sorption isotherms.

An understanding of sorption parameters is therefore extremely valuable and it can indicate the possible behaviour of packaging films under different relative humidities (RH). The knowledge of sorption isotherms at different temperatures enables an evaluation of heat of sorption, which determines the interaction between water molecules and film forming material.

The aim of this present research is to investigate equilibrium water vapor sorption behaviour of sago starch based films in terms of isotherm parameters and isosteric heat of sorption so that moisture barrier efficiency of this packaging film can be evaluated.

## Materials and Methods

### *Materials*

Granules of sago starch were received from a local merchant and used as received. Other chemicals such as glycerol (GY) and various salts were purchased from High Media Laboratories, Mumbai, India. Double distilled water was used throughout investigations. Saturated salt solutions of different salts were prepared to provide a wide range of relative humidities (RH) as described elsewhere [14].

### *Preparation of sago starch (SS) films*

SS film was prepared by microwave induced gelatinization of sago starch followed by evaporation of solvent. In brief, 1 g of sago starch was added into 20 ml of distilled water at $70^0$C followed by addition of 0.25 ml of glycerol. The total volume of colloidal dispersion, so obtained, was made up to 25 ml by addition of appropriate quantity of water. The colloidal dispersion was put in microwave oven (LG, model MS-1947C) and irradiated at 640 Watt for 30 seconds, which yielded an almost transparent solution. The solution was poured into Petridish and put in an electric oven (Tempstar, India) at $50^0$C for a period of 24 hr. Finally the film was pealed off and kept in a dessicator for further use.

### *Characterization of film*

*FTIR – Spectral analysis*

The FTIR Spectra of glycerol plasticized sago starch film was recorded on Schimadzu 8400 S Fourier Transformation Infrared Spectrophotometer using KBr.

*XRD analysis*

XRD analysis was performed with a Miniflex II desktop Xray Diffractometer (Japan).

### *Equilibrium moisture sorption studies*

The moisture sorption isotherms were determined gravimetrically using the static method as described by Alhamadan *et al* [15]. Constant relative humidity (RH) atmospheres were obtained with saturated salt solutions (LiCl, $CH_3COOK$, $K_2CO_3$, NaBr, NaCl, KCl, $BaCl_2$, $K_2SO_4$ and $H_2O$) covering a water activity range from 0.10 to 1.0 at desired temperature. The pre-weighed dried samples, in triplicate, were placed inside each of the eight desiccators containing the saturated salt solutions. The samples were weighed periodically (every day for a period of seven days) until the percentage of sample mass, changed between two successive measurements, was less than 1%. The moisture adsorbed by samples was calculated using the following expressions:

$$\text{Equilibrium Moisture Content (EMC)} = \frac{W_e - W_o}{W_o} \text{ (g/g film)} \qquad \text{..... (1)}$$

where $W_o$ and $W_e$ are weights of the film in the initial and equilibrated state respectively.

### *Analysis of moisture update data*

Several models have been proposed to correlate the equilibrium moisture contents to relative humidity of environment [16], which can be divided into several groups; kinetic model based on a

multilayer and condensed film (GAB model), semi-empirical Halsey and Henderson model and purely empirical Oswin model. In this study, all the four above mentioned models were applied on the experimental moisture uptake data. A brief description of these models has been given in Table 1. Nonlinear regression was used to fit the models and estimate the GAB isotherm constants, while Oswin and Halsey and Henderson models were evaluated by linearized forms of equations using SPSS for windows version 6.0. The suitability of the isotherm equations was evaluated and compared using the mean relative deviation modulus (MRD) as prescribed by Wang and Brenn [17].

$$MRD = \frac{100}{N}\sum_{i=1}^{N}\frac{X_{ei} - X_{pi}}{X_{ei}} \qquad \text{.....(2)}$$

Where $X_{ei}$ = measured equilibrium moisture content (g/gdry basis) ; $X_{pi}$ = predicted equilibrium moisture content; and N = number of data points

**Table 1. Different sorption models fitted to the equilibrium moisture uptake data.**

| | | |
|---|---|---|
| **GAB** | $Mc = M_oCKa_w/[(1-Ka_w)(1-Ka_w+CKa_w)]$ | [18] |
| **Oswin** | $Mc = A[a_w/(1-a_w)]^B$<br>$lnMc = lnA+Bln[a_w/(1-a_w)]$ | [19] |
| **Halsey** | $Mc = (-A_1/lna_w)^{1/A_2}$<br>$ln\ Mc = lnA_1+A_2ln(-lnaw)$ | [20] |
| **Henderson** | $ln\ [-ln(1-a_w)] = ln\ K + n\ ln\ Mc$ | [21] |

Nomenclature: $M_c$ =equilibrium moisture content (g $g^{-1}$ dry basis), $M_0$ = monolayer moisture content (g $g^{-1}$ dry basis); $a_w$ = water activity; A and B = Oswin constants, $A_1$ and $A_2$ = Halsey constants ; K and C are GAB constants, n and K are Henderson constants.

***Thermodyanamic properties***

The application of thermodynamic principles to sorption isotherm data has been used to obtain more information about the properties of water, food microstructure and physical phenomena on the film surfaces and sorption kinetic parameters. The net isosteric heat of sorption ($q_{st}$) is defined as the total heat of sorption ($Q_{st}$) minus the heat of vaporization of water at the system temperature [22]. The net isosteric heat of sorption or differential enthalpy shows the energy requirement for removing moisture from food material (water - solid binding strength) and has a practical use in complete drying calculations and modelling of energy [23]. Moreover, entropy change also plays an important role in the energy analysis of food processing systems and the differential entropy of sorption ,$S_d$ ,can be calculated using Gibbs- Helmholtz equation [24].

For calculation of $q_{st}$ and $S_d$, various values of moisture content from 0.18 to 0.45(g $g^{-1}$ dry basis) were used. Through these values, the values of $a_w$ were determined at different temperatures. By plotting $lna_w$ versus 1/T, for a specific moisture content of the material and then determining the slope ($-q_{st}/R$) and intercept ($S_d/R$), the net isosteric heat ($q_{st}$) and differential entropy ($S_d$) of sorption were obtained [25] using the following equation:

$$-lna_w = \frac{q_{st}}{R}.\frac{1}{T} - \frac{S_d}{R} \qquad \text{.....(3)}$$

where $q_{st}$ is the net isosteric heat of sorption (kJ $mol^{-1}$), $S_d$ is the differential entropy (kJ $mol^{-1}K^{-1}$), R the universal gas constant (kJ $mol^{-1}$ $K^{-1}$), T is the absolute temperature, (K) and $a_w$ is the water activity. The compensation theory proposes a linear relationship between $S_d$ and $q_{st}$ [26]

$$q_{st} = T_\beta . S_d + \alpha$$

The isokinetic temperature ($T_\beta$) and constant $\alpha$ were calculated using linear regression. In order to corroborate the compensation theory, a statistical analysis test is provided by Knrg *et al* [27]. The harmonic mean temperature $T_{hm}$ was given as follows:

$$T_{hm} = \frac{n}{\sum_1^n (1/T)} \qquad \text{.....(4)}$$

The compensation theory only applies if $T_\beta \neq T_{hm.}$ If $T_\beta > T_{hm}$ then the process is enthalpy driven, while if $T_\beta < T_{hm}$ the process is considered to be entropy controlled.

## Results and Discussion

### *FT-IR spectral analysis*

FT-IR analysis of glycerol plasticized sago starch film in Figure 1 shows peak of OH – group stretching at 3576$cm^{-1}$, absorbance near 2960 $cm^{-1}$ and 1485 $cm^{-1}$ is due to C-H stretching and bending vibrations, absorbance band between 1200 $cm^{-1}$ and 1000 $cm^{-1}$ is due to C-O stretching.

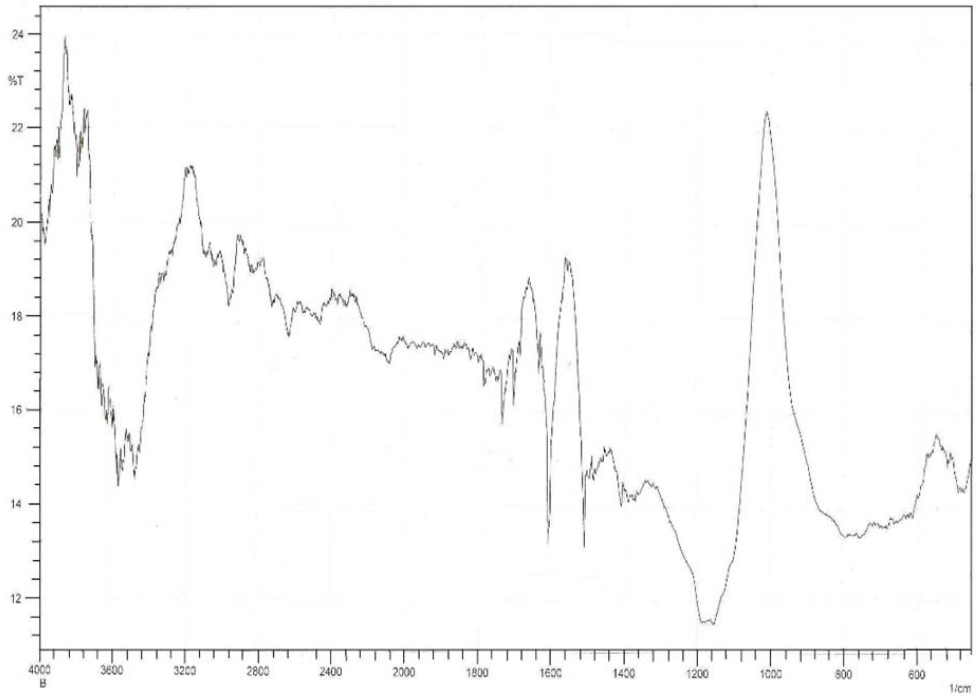
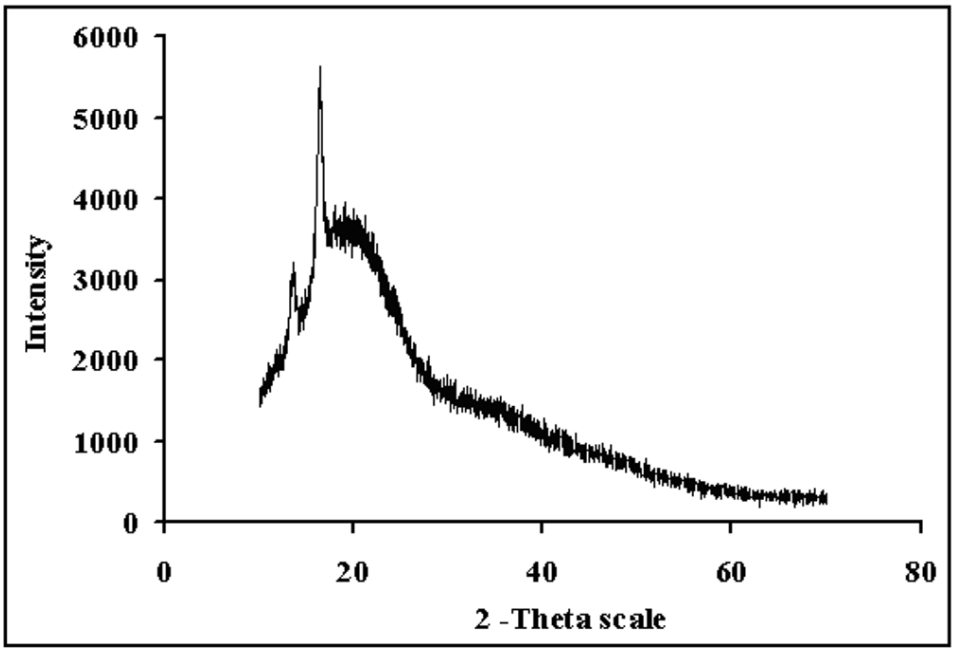



**Figure 1. FTIR of glycerol plasticized film.**

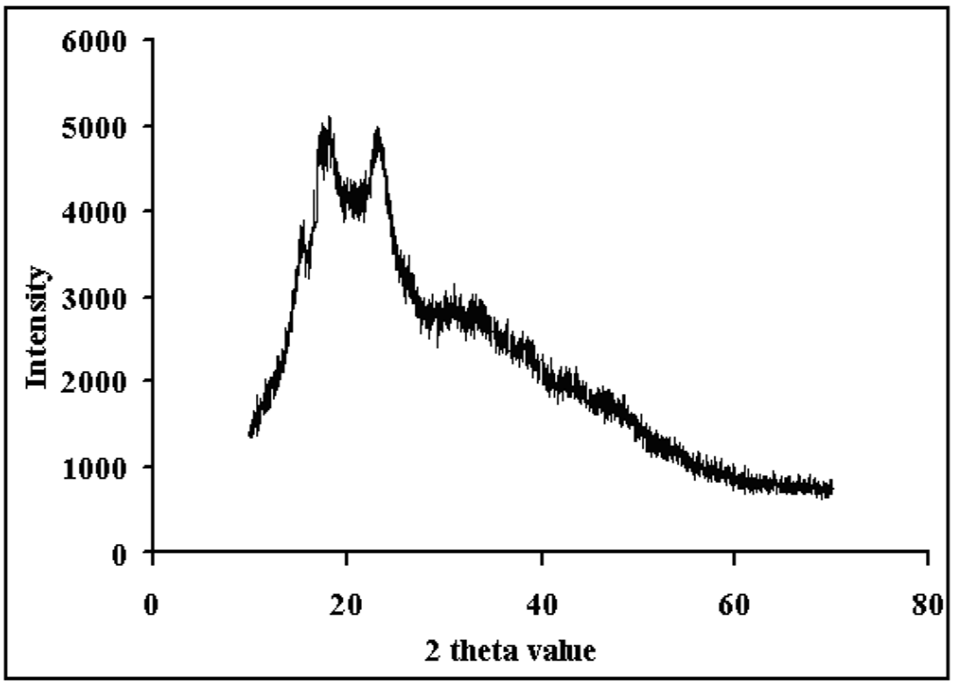
### *XRD analysis*

The X-ray diffraction patterns of (a) plain sago starch film and (b) sago starch powder are shown in Figures 2(a) and (b) respectively. As can be seen, the sago starch powder shows typical A type diffraction pattern with strong reflections at $15.1^0$, $17^0$, $17.5^0$ and $23^0$ [28]. It also evident that original crystalline structure is slightly modified in the film elaboration process. The film exhibits

two reflection peaks, one at $15.1^0$ and the other at $17.2^0$. An increase in peak intensity and decrease in width indicate enhanced crystallinities in the film. This may be due to fact that starch molecules in the dissolved state have enhanced chain mobility and this allows possibilities of their rearrangement in networking during film formation process. The amorphous zone, present in both the diffractograms is mainly due to amylopectin [29].




2(a)




2(b)

**Figure 2. X-ray diffraction pattern of (a) plain sago starch film and (b) sago starch powder.**

***Sorption isotherm***

The experimental moisture uptake data for sago starch based film at 23, 31 and 37$^0$C are shown in Figure 3. All curves were typical sigmoid in shape, characteristic of type II isotherms, which are typical of most biopolymer materials such as cellulose [30], starch [31] and HPMC [32], etc. The sorption isotherms demonstrate an increase in equilibrium moisture content with increasing water activity at constant temperature. At low and intermediate water activities, the so-called multilayer sorption region, moisture content increases linearly with $a_w$, whereas at higher water activity levels, the so-called capillary condensation region, water content rapidly increases with $a_w$. This behaviour may be attributed to the fact that, at low water activities physical sorption occurs on strongly active binding sites of substrate, such as –OH groups present on the surface of film. In the intermediate $a_w$ range sorption takes place at less active sites. Moreover, for a given water activity the equilibrium moisture uptake is observed to decrease as the temperature is raised from 23 to 37$^0$C. This behaviour is most common for hydrated food materials and may simply be attributed to the fact that increase in temperature causes an increase in kinetic energy of water vapor molecules and so van der Waals forces between sorbed water vapor molecules and starch film become weak, thus resulting in less moisture uptake. Thus moisture sorption may be regarded as an exothermic process. Similar results have also been reported in our previous work on chitosan–based edible films.

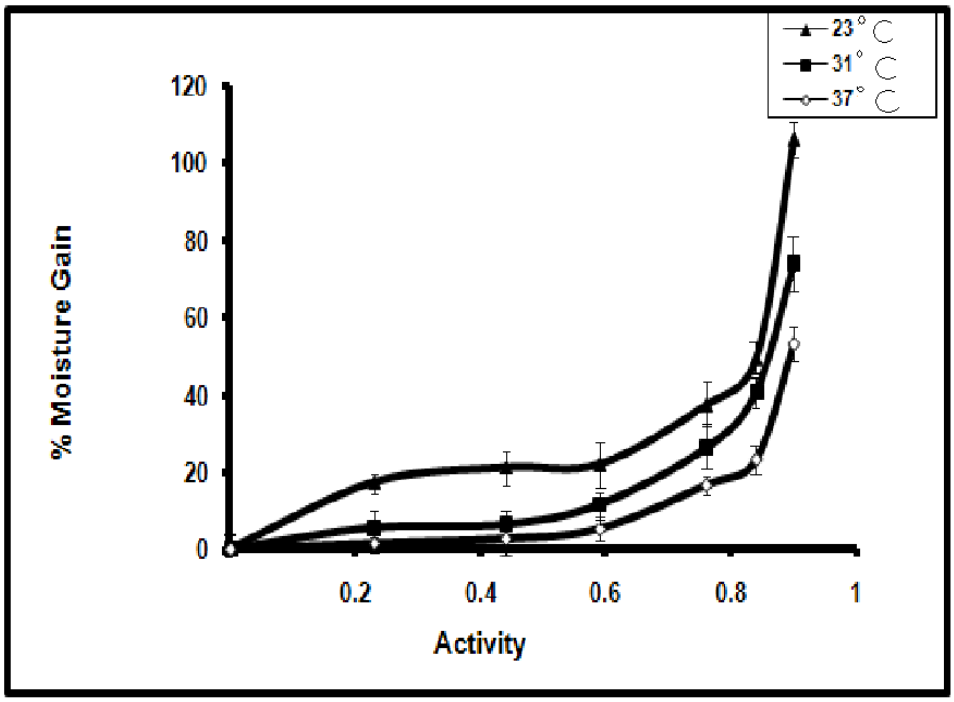



**Figure 3. Equilibrium moisture uptake of sago starch films as a function of water activity at different temperatures.**

The suitability of various isotherm models, shown in Table 1, was tested on the equilibrium moisture uptake data shown in Figure 3. The polynomial curves for GAB model and linear plots for Oswin, Halsey and Henderson models were obtained, as shown in Figures 4, 5, 6 and 7 respectively. Based on % MRD values, the order of fitness of these models was Halsey>Oswin>GAB>Henderson. The related parameters of these models are shown in Table 2. As stated earlier, GAB model is theoretically sound, whereas other models are empirical/semi-empirical in nature. Therefore, parameters of GAB isotherm need some discussion. The value of the monolayer moisture content ($M_0$) is of particular interest since it indicates the amount of water that is strongly adsorbed to specific sites at substrate surface and is considered as the optimum value to assure stability of substrate material [33]. Therefore, $M_0$ is recognized as the moisture content affording the longest time period with minimum quality loss at a given temperature. Below it, rates of deteriorative

**Table 2. Related parameters of the models.**

| Model | Constants | Temperature ($^0$C) | | |
|---|---|---|---|---|
| | | 23 | 31 | 37 |
| GAB | Mo | 0.08923±.0039 | 0.0393±0.0128 | 0.0216±0.0117 |
| | C | 8.7333±0.0139 | 162.762±1.0043 | 2.884±0.0386 |
| | K | 0.9956±0.0114 | 1.0729 ±0.0124 | 1.0903±0.01156 |
| | % MRD | 9.21 | 4.88 | 3.01 |
| | $R^2$ | 0.9038 | 0.9176 | 0.9602 |
| Halsey | $A_1$ | 0.1824±0.01133 | 0.0656±0.01131 | 0.0231±0.01138 |
| | $A_2$ | -0.6547±0.0106 | -1.0437±0.0129 | -1.3843±0.0122 |
| | % MRD | 6.62 | 3.28 | 2.58 |
| | $R^2$ | 0.9065 | 0.9768 | 0.9924 |
| Oswin | A | 0.2465±0.0117 | 0.1052±0.01183 | 0.04307±.0036 |
| | B | 0.4984±0.0121 | 0.8047±0.0133 | 1.0766±0.0120 |
| | % MRD | 6.28 | 5.06 | 4.12 |
| | $R^2$ | 0.8585 | 0.9489 | 0.9811 |
| Henderson | n | 1.047±0.0361 | 0.732±0.0311 | 0.573±0.0347 |
| | K | 2.97427±.0033 | 3.4178100±.0039 | 3.91180±.00346 |
| | % MRD | 5.20 | 8.91 | 4.62 |
| | $R^2$ | 0.770 | 0.889 | 0.942 |

reactions are minimum. Hence at a given temperature, the safest water activity level is that corresponding to $M_0$ or lower. The values of monolayer content $M_0$, as shown in Table 2 are 0.08923± 0.0039, 0.0393± 0.0218 and 0.0216± 0.0117 at 23, 31 and 37$^0$C respectively. The $M_0$ values for pinhao (*Araucaria angustifolia* seeds) starch are 0.10, 0.08 and 0.07 kg/kg dry basis at 20, 30 and 40$^0$C respectively as reported by Thys *et al* [34]. These values were in quite good agreement with those reported in this present study. The $M_0$ values show a decreasing trend with increase in temperature. This may be attributable to the decrease in number of active sites for water binding because of physico-chemical changes in starch induced by temperature. Also, according to Palipane *et al* [35] another possibility could be that with an increase in the temperature, the water molecules become activated due to an increase in their energy level, causing them to become less stable and to break away from the water binding sites of substrate materials.

Another GAB constant C describes adsorbent - adsorbate interactions and according to Blavohec [33] the parameter C should fulfill the following relations: for C>2 the GAB model should yield a sigmoidal shape curve with point of inflection (type II of Brunauers (1943) classification): and for 0<C<2 the isotherm is of the type III only (isotherm without point of inflection). In this study the value of C was greater than 2 for all temperatures studied and the isotherm curves obtained were also sigmoidal, thus supporting the above predictions. Finally, the value of K provides a measure of the interactions between the molecules in multilayer with the adsorbent and tends to fall between the

energy values of the molecules in the monolayer and that of liquid water. The prescribed range for K values is 0 <K≤1. As can be seen, the values of K, obtained at 23, 31 and 37$^0$C fall within the prescribed range. However, the observed value of unity (i.e. K=1), is indicative of the fact that multilayers have properties of liquid water [36].

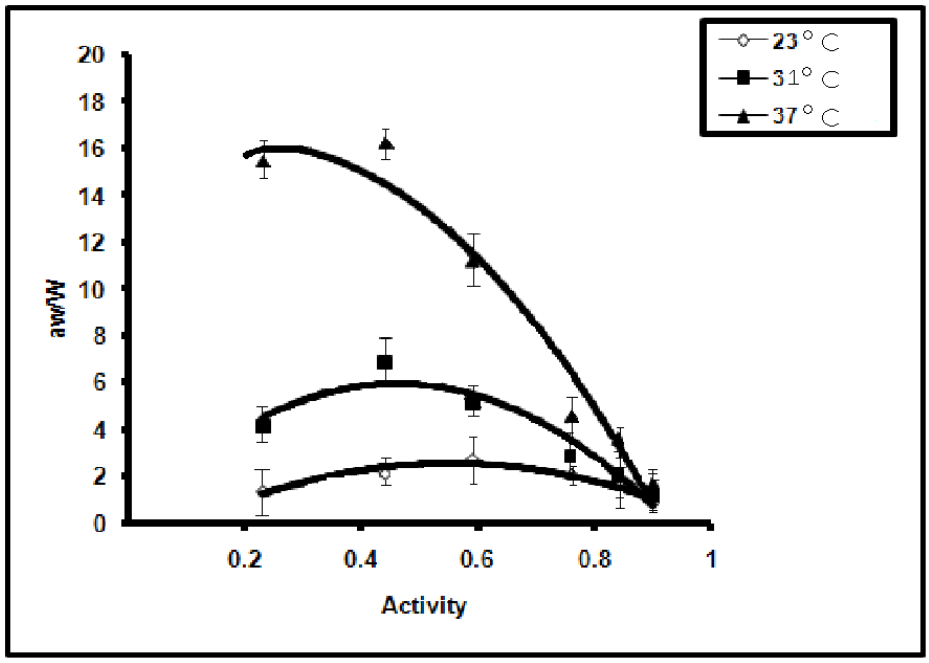



**Figure 4. Polynomial curves obtained between $a_w$/W and $a_w$ for starch films at three temperatures.**

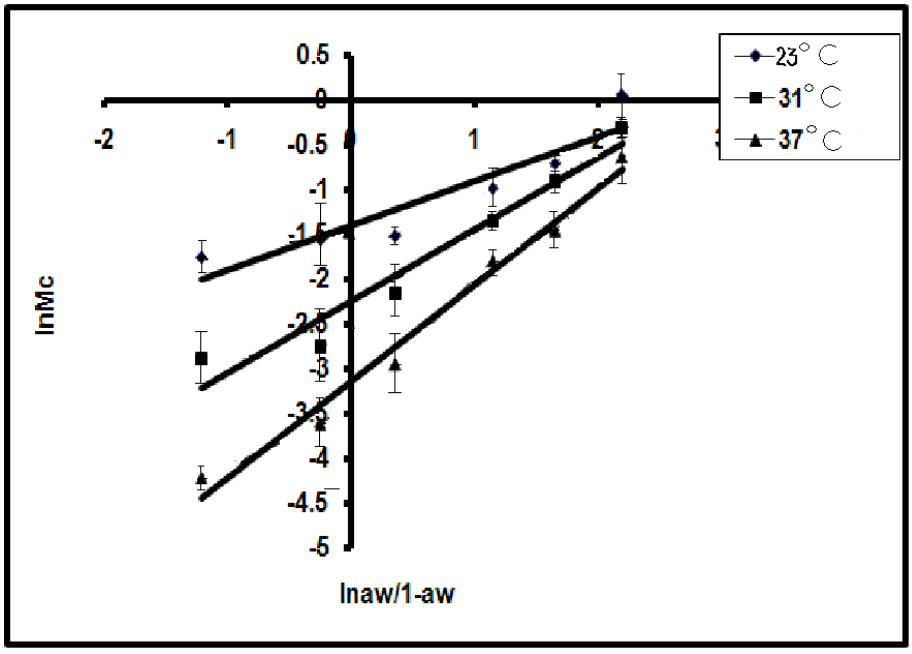



**Figure 5. Linearized plots of Oswin isotherm model for moisture sorption data at different temperatures.**

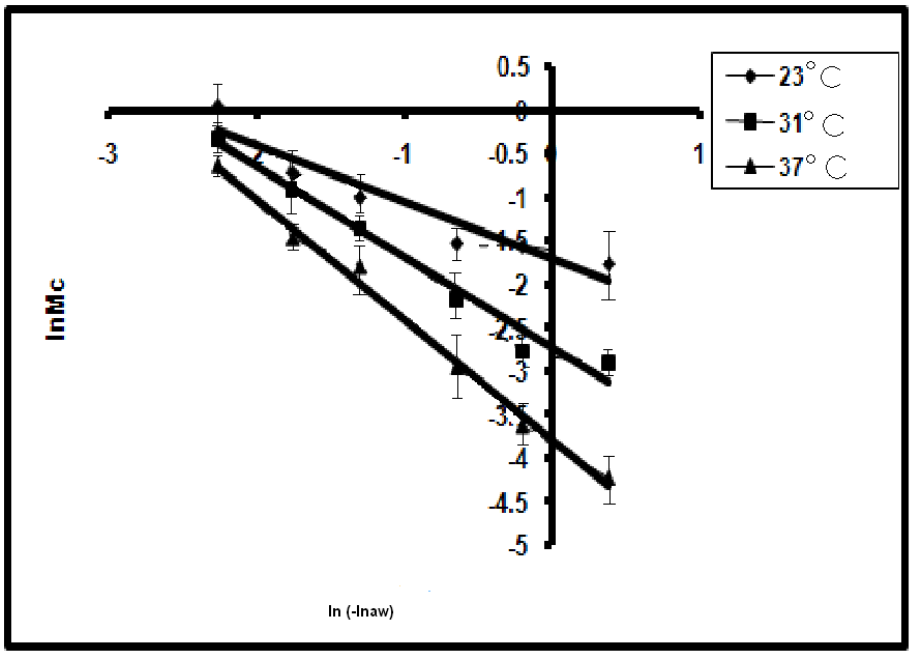



**Figure 6. Linearized plots of Halsey isotherm model for moisture sorption data at different temperatures.**

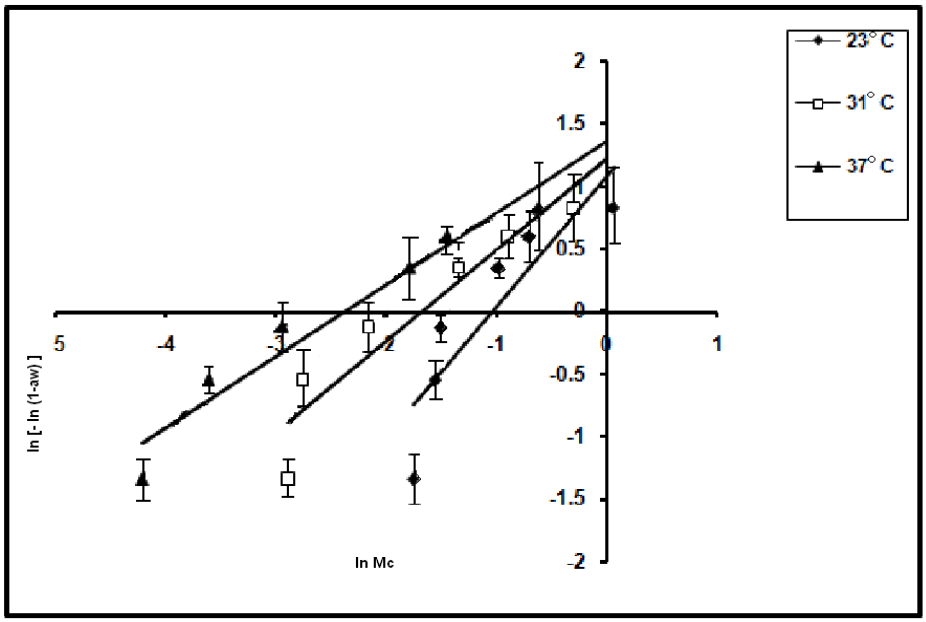



**Figure 7. Linearized plots of Henderson isotherm model for moisture sorption data at different temperatures.**

***Evaluation of thermodynamic parameters***

In order to determine net isosteric heat of sorption $q_{st}$ and differential entropy of sorption $S_d$, water activities at three temperatures, namely 23, 31 and 37$^0$C, were determined for a definite moisture content range of 0.18 to 0.45 g/g dry basis using the sorption isotherm data displayed in Figure 3. Then plots were obtained between ln $a_w$ and 1/T for each moisture content value selected, as shown in Figure 8. The respective $q_{st}$ and $S_d$ values were obtained using slopes and intercept of linear plots.

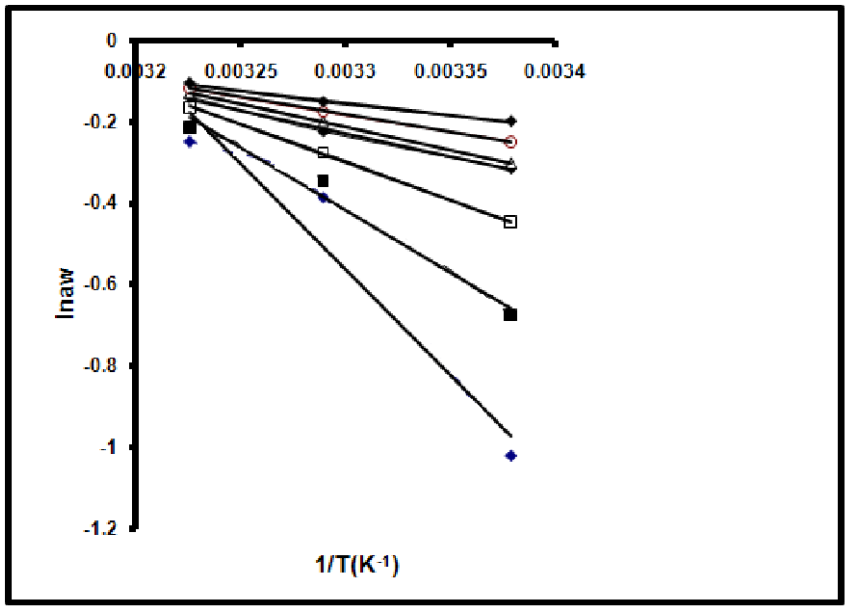



**Figure 8. Plots of $\ln a_w$ versus 1/T for determination of $q_{st}$ and $S_d$.**

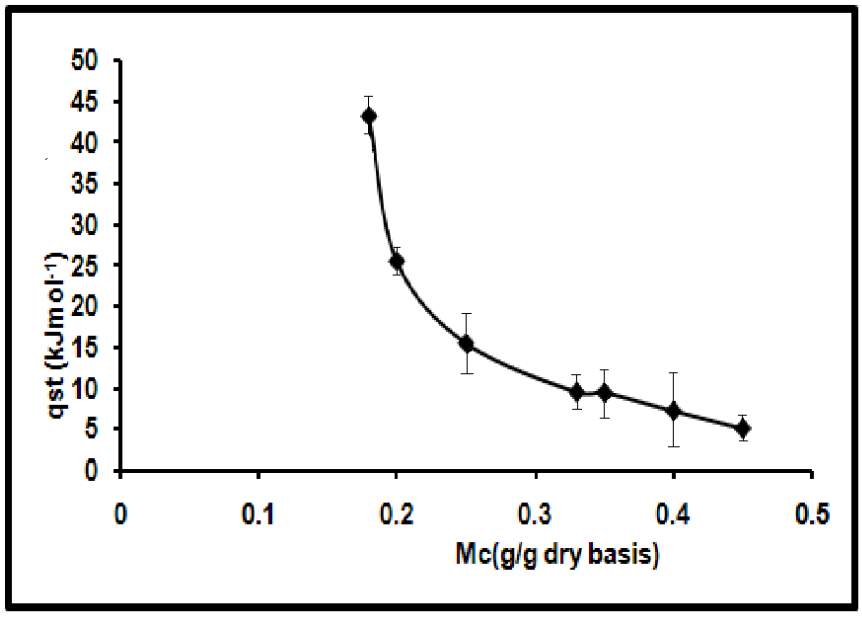



**Figure 9. Variation of $q_{st}$ with moisture content.**

Figure 9 shows that net isosteric heat of sorption decreases with the equilibrium moisture content. This decrease can be attributed to the fact that initially sorption occurs at the most active sites, thus giving rise to greatest interaction energy. As the moisture content increases, the sites available for water vapor sorption decreases, thus resulting in lower values of $q_{st}$ [37]. At low moisture content higher $q_{st}$ values could be due to strong interaction between water molecules and hydrophilic groups of starch molecules. Almost similar results have also been reported by Al-Muhtaseb *et al* [38] who studied the water sorption and thermodynamic properties of starch powder. The differential entropy of sorption $S_d$ versus moisture content plot is also shown in Figure 10. It is clear that differential entropy also decreases with increasing moisture content. These results show strong dependence of net isosteric heat of sorption $q_{st}$ and differential entropy of sorption $S_d$ on moisture content. Similar trends have been also reported elsewhere [39].

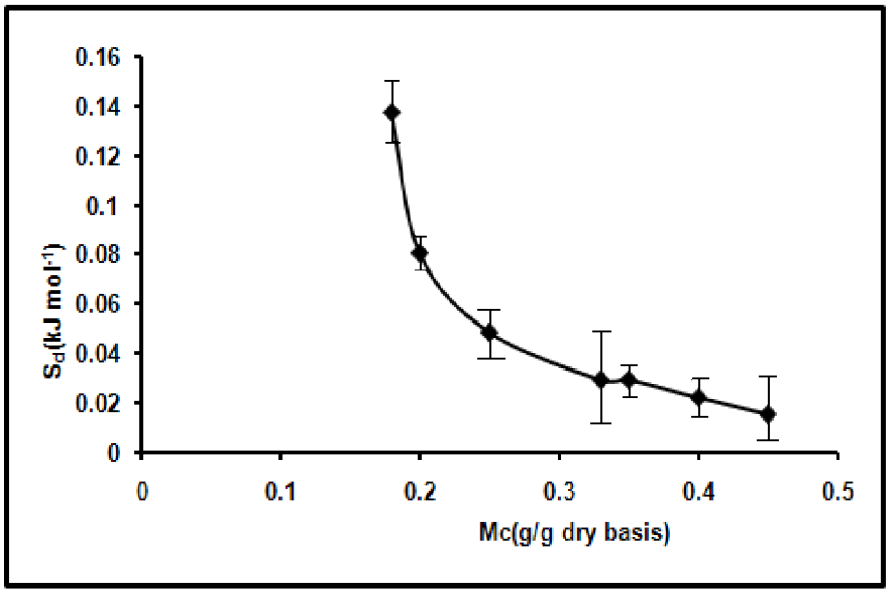



**Figure 10. Variation of $S_d$ with moisture content.**

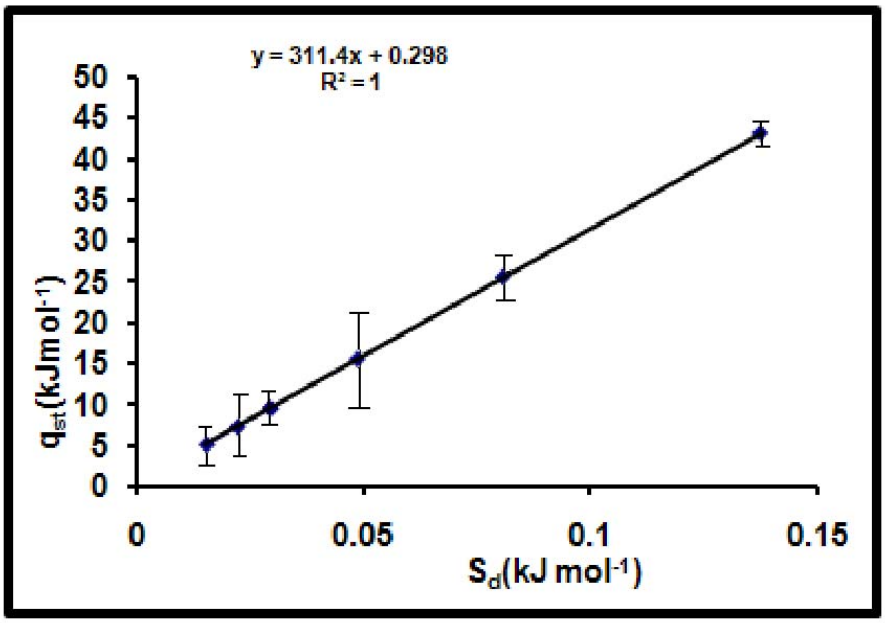



**Figure 11. Plot between $q_{st}$ and $S_d$.**

***Entropy–enthalpy compensation theory***

The plot of $q_{st}$ versus $S_d$ shows a linear relationship for sago starch based film as shown in Figure 11. The values for the parameters $T_\beta$ and $\alpha$, calculated from the data by linear regression, were found to be 311.4 and 0.298 respectively ($R^2$ =1). The isokinetic temperature ($T_\beta$) is the temperature at which all the sorption reaction will take place at the same rate. The value of $T_\beta$ (i.e. 311.4 k), found in the present work appears to be quite low as compared to the value of 428, reported by Thys *et al* (2010) for pinhao starch. For adsorption isotherms of starch materials McMinn *et al* (2005) found $T_\beta$ values between 366.8 and 466.8k. The harmonic mean temperature was also calculated using equation (4) and was found to be 303.2 k. It is clear that two temperatures, namely $T_\beta$ and $T_{hm}$ are not equal but differ slightly. As the values of $T_\beta$ and $T_{hm}$ do not differ appreciably, this is an indication that the compensation theory is not applicable in the present study.

## Conclusion

The uptake of moisture by sago starch based film was studied at three temperatures and the data obtained was interpreted in terms of Oswin, GAB, Halsey and Henderson isotherm models. The isosteric heat of sorption ($q_{st}$) and differential entropy ($S_d$) tended to decrease with increasing moisture content. The moisture sorption process appears to be enthalpy driven ($T_{\beta} > T_{hm}$).

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