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Evaluation of Moisture Absorption and Permeability to water vapor Cellulose Nanocomposites and its effect on Moisture, Mold and Yeast Packed with Walnuts

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ABSTRACT: Given the importance of technology in improving the efficiency of Nano food packaging and storage of goods according to the walnut is an important and strategic Product for country, the effect of moisture absorption and permeability to water vapor cellulose nanocomposites on moisture and mold and yeast in packed walnuts were studied. The film was prepared based on CMS film and nanocomposite Carboxymethyl cellulose / polyvinyl alcohol-clay and characteristics such as permeability to water vapor and moisture were evaluated. The moisture results and mold and yeast in Walnut were analyzed. The results showed that the water vapor permeability was significantly reduced and the g / MhPa 0.036 control film g / mhPa 0.029 for movies with3% Nano clay concentrations decreased. The product features with 0.5 percent reduction compared to control moisture than Carboxymethyl cellulose / polyvinyl alcohol – clay cover due to maintaining the nutritional value at the end of the maintenance period is shown. With the passage of time (90 days) in all treatment, mold count was significantly ($P < 0.05$) reduced. Although this decline was greater in the treatment contains 3% nano so that at the end of the period it had the least amount of molds and yeasts (log cfu / gr 0.17 ± 3.4). So coatings in terms of mechanical and chemical properties for packaging nuts were evaluated positive. But in addition to achieving these desirable characteristics, covering was an appropriate factor for preventing mold growth during storage time was relative.

Keywords: moisture, water vapor, mold and yeast, walnut kernel, cellulose nanocomposites.

INTRODUCTION

Film or coating as a layer of polymer compounds, is placed on the food. The coating is the film that is used for packing food. They are based on natural polymers or synthetic structure with special properties. They create a barrier against the transfer of materials (water, gas and oil), preservation and transfer of food ingredients and additives and colors, flavors, prevent the growth of microorganisms on food surfaces and mechanical protection. Functional properties of the film are very much affected by the cause of such formulations, technology, film, solvents and additives. The use of films and coatings is mainly due to their potential to provide compounds of dam's moisture, oxygen, taste and smell, for food quality and increase in shelf life. So permeability of the film should be set for such cases.

Permeability characteristics that influence the extent to which a substance is dissolved And then spreads with a thrust of the film related to the influence of the concentration differences on both sides of the film, (Gnadyvs, 2002). For many food applications, the most important feature is the film or coating against moisture transfer. Because in many food a certain level of water activity must be maintained and destructive chemical and enzymatic reactions activity are strongly influenced by water or moisture content. So moisture transfer in the food products highly affects quality, stability and safety. Changes in the moisture content of the food can occur between or within food and the atmosphere around it. Moisture can also move be due to capillary forces and surface diffusion. Moisture transfer speeds between and around food is reduced by food packaging film or coating.

In addition to water vapor, gases transfer such as oxygen and carbon dioxide specifically affect the stability of food. The initial state of corruption in many foods, including oxidation of fats, vitamins, pigments and aromatic compounds (Kstr and Fnma, 1986). Establish and enhance quality has a direct relationship with increased durability and improved safety. With protective coating durability increases and the possibility of contamination with foreign materials is reduced. Recently, the demand for fresh and processed foods increased. Therefore, the need for food products with high durability and safety is felt. The use of renewable bio material in food packaging looks important. Because it reduces final consumption of synthesis packaging materials (Zhang and Gnadyvs, 2006). Nanotechnology has known as revolutionary in many industries including food packaging and processing (Cho *et al.,* 2007). And its use as food packaging materials at the nanoscale is the most applications in the field of food (Bvmstr *et al.,* 2009). Nanocomposites are new methods alternative to traditional methods for improving the properties of polymers (Arora *et al.,* 2009). The definition of composites and nanocomposites can be cited like this that composite is a mixture of polymers and filling material and nano-composite is a composite with at least one dimension filling (length, width or thickness) in the the nanoscale (1 to 100 nm). Nanocomposites polymer compared with conventional composites, have better interaction between the polymer matrix and filler. Uniform distribution of the nanoparticles in a polymer matrix increase nanoparticles to the surface area matrix that improves mechanical, heat and prohibitive properties (Chvdalakys, 2009). Biopolymer due to poor mechanical and inhibitory properties is limited that it may be developed by adding components (fillers) form additional compounds. Most of filling materials have weak interaction with matrix that by reducing the size of filling dimension has a tendency to expand. . Nanoparticles have larger surface area than their microbial scale counterparts that are favorable for the interaction of the filling - matrix and the matter yield. In addition to the ability of nano, nano particles can be added to polymer when it has other functions such as antimicrobial activity, immobilization of enzymes, bio-sensors, etc. (Hnryt and Zrdv, 2009). In recent years, one the most used nano-particles that is widely used in the packaging industry along with a variety of polymers derived from oil (plastics) as well as a variety of biodegradable polymers in order to modify the physical properties, mechanical and chemical is, nano clay. The application of nanotechnology to existing polymers may be new possibilities to improve not only the features but also cost efficiency (Sorrentino *et al.,* 2007). Clay nanoparticles are usually considered two-dimensional pages with minimal thickness (about nm). When these pages are dispersed in a polymer matrix, creating a path that greatly reduces the transmission of gas. Two special features that play an important role in the production of nano-clay nanocomposites include one opening layers from each other and dispersed in a polymer matrix and modifying surface for better interaction between the polymer matrix and nanoparticles (Chvdalakys, 2009). With layers of nanoclay in the fields of biopolymer, film inhibition against gaseous molecules such as oxygen, water vapor and aroma compounds increases. Because on the one hand, local mobility of the polymer chains are reduced and space between them become less and on other side gases to pass through the polymer must travel a long Zigzag way. Thus, the time it takes to pass through the molecules of the film increases and therefore the permeability of the film is reduced (Svsvrnvyt et al., 2010). carboxymethyl cellulose or CMC is cellulose derivatives that is produced by reacting cellulose with sodium hydroxide and acetic chloro acid. It's a matter that is obtained of being substituted carboxymethyl groups (-CH_2-COOH) instead of some of the hydroxyl groups (-OH). Cellulose because of its chemical structure is crystalline and insoluble in water but carboxymethyl cellulose is soluble and forms a strong flexible film (Dbvfvrt, 1997). The carboxymethyl cellulose is one of the cheapest biopolymer (biopolymer) that is produced industrially (Pena and Torres, 1991). Polyvinyl alcohol (PVA), is the most common synthetic water-soluble polymer that is produced from polymer of vinyl acetate monomer to polyvinyl acetate and then hydrolysis of, a polyvinyl alcohol. Polar nature and solubility in water, non-toxic, high tensile strength and good adhesion properties and emulsifying properties has made this compound suitable for use in composites containing natural polymer (Abolqasemi *et al.,* 2010). World's largest Synthetic polymers produced in order to abundance, is PVA. Biodegradation in the environment is the most important feature (Ramaraj, 2007). Persian walnut plant is one of the world's valuable resources, especially Iran. Iran include a large part of the Central Asian region as a center of diversity and the emergence of many species, especially species of Persian walnut (Ford, 1975). Walnut production is in third place after the US and China, but it has only half a percent of global trade (Zayvyng and Yufei, 2012). That if producers do greater emphasis on increasing the quality of the products they export more.

MATERIALS AND METHODS

Film preparation: 3.5 g of CMC in 200 ml of water was dissolved and was heated with stirring for 45 minutes at 90°C. With 10% PVA v / v, CMC separately in 50 ml of water was heated at 90 ° C for 40 minutes.

Percent of zero, 5.0, 1 and 3 percent of nanoclay (w / w CMC) in 100 ml was spread in distilled water and processed for 10 minutes exposed to ultrasound. Ultrasound can modify the properties of the films (Yuki and Ishikawa, 1976 and Kstr and Fnma, 1989). CMC solution and nano clay were mixed together and headed for 15 minutes at 65°C with stirring. Then PVA solution was added and stirring continued for 30 minutes. 1.4 mL of glycerol (40 ml per 100 g of CMC) was added to the solution and stirring continued at 65°C for 20 minutes. This solution is cooled to ambient temperature and film-forming solutions are poured into a glass jar and were dried at 55°C for 18 hours. And dried films were isolated from the surface (Abolqasemi Fakhri *et al.*, 2010). Walnut kernels were packed in produced coatings.

Determine the amount of film moisture: To measure the amount of moisture absorption Dafnz and Angel method (2000) was used. Examples of films with dimensions of mm 0.3 × 23 × 23 were prepared in the desiccator containing calcium sulfate for 24 hours. After the initial weighing samples containing were transferred to saturated desiccating solution of sodium chloride at a temperature of 20-25°C. At different times the weight of the samples were measured to constant weight. The amount of moisture absorption is calculated using the following equation:

$$\text{Moisture absorption (\%)} = \frac{Wt - W0}{W0} \times 100$$

W_0 the initial weight of samples

W_t Sample weight after time *t*, at the7 5% relative humidity

Determine the amount of water vapor permeability (WVP): The amount of water vapor permeability of the films were presented in accordance with standard ASTM E-996-00 and was measured in three replications (ASTM, 1995). Basis of test is gravimetric method and to conduct this test glass cup with internal diameter of 3 cm and a height of 3.5cm was used. In each of the cups, 8 ml of distilled water (to 100% relative humidity) was poured. Sample movies were established on opening of little cups by grease, rubber gasket and metal clamps and little cups were placed within desiccators containing silica gel (0% RH). Every 2 hours little cups are weighed until constant weight of two sequential weighing, and the weight loss is determined using a digital scale with resolution 0.0001. Weight changes over time for determining the rate of the water vapor transmission among film (the Permeability to water vapor) is calculated according to the formula below.

$$WVP = \frac{\Delta m \times d}{A \times \Delta t \times \Delta P}$$

wvp Permeability to water vapor (g / Mtr.sanyh. Pascal) Weight loss of the cup (G, A exposed level (7.06), time (s), D is Thickness (m) and the partial pressure difference between the inside and outside of the cup. The calculated amount of pressure difference on both sides of the film (relative humidity of 100% and 0%), was considered according to the reference tables, 3.179 Pascal (using saturated steam table).

Packaging Walnut: Walnut kernels were selected according to National Standard No. 18 (19) of the 700-hectare garden of Shahmirzad in Semnan province, and were packaged by carboxymethyl cellulose / polyvinyl alcohol films with a concentration of 0.5, 1 and 3 percent nano-clay. Walnut kernel packed with movie without clay was used as a control. The condition of samples was at 25 ° C (room temperature) and relative humidity environment was for 90 days.

Determine the moisture content of the product: To measure the moisture content, the glasses were weighing, after drying in the oven and cooled in the desiccators. Then a few grams of sample was weighed and put in the dish and sample weight was recorded. The 70-liter containers made of Behdad Company with the sample were placed in an oven at 105°C for 24 hours. After 24 hours, the Petri dishes with samples were removed from oven and re-weighed in desiccators. Moisture content was determined by calculating the difference between the weights (AOAC, 2005).

$$\text{Moisture content} = \frac{(\text{initial weigh} - \text{final weigh})}{(\text{G}) \text{ initial weight}} \times 100$$

Study of biological characteristics: Microbial counting method includes standard total count and mold count technique. (AOAC, 1984). For microbial analysis at first decimal dilutions of food samples tested, are prepared.

The empty container and sterilized blender, and exactly 10 grams of uniformed sample are weighed within it. 90 ml saline diluent is added and by shaker uniformed mixture of sample with dilution1.0 is achieved. For preparing 0.01dilution tube containing 9 mL of saline, 1 mL of 1.0 dilution was added and for preparing dilution 0.001, a tube of 9 ml of saline, and 1 ml of 0.01 dilution is added. For microbial testing in this study we used of 0.01 and 0.001 dilution (Karimi,1996).

Preparation medium: 15/8 g DG 18 media with 500 ml of water is mixed put on the fire until it is dissolved completely. Then we add 17.38 grams of glycerol. Culture medium will be in autoclave for 15 minutes at 121°C and when temperatures reach 55 degrees, and then we add 5.0 grams of chloramphenicol. After ensuring complete dissolution we pure them in plate and put in refrigerator for 18 hours.

The cultivation of the product and the total count: After closing and hardening of the medium, 1 ml of different dilutions that is uniformed by Shaker, using the Micro Sampler 1.0, is poured in plate. And by using sterile shovel dilution is spread on our culture. All this was performed under the hood of microbial (Beasat). Colonies are counted after 3 days in the incubator (company Behdad, making Iran) at 25°C. Total count with three replications was conducted. Data from visual counting plates is multiplied on dilution used in the image and we split weight of the sample, and the number is taken from the log. Logarithm of the number of colonies obtained per unit weight (Salam, 2004).

Statistical methods of data analysis: In order to analyze the data, and the data obtained different testing of completely randomized design was used. In order to determine the difference between the average number (three times for 4 treatments: control, 0.5%, 1% and 3% clay) one-way ANOVA analysis for film properties and two way to measure the properties in packed Walnut was used. At all stages, statistical analysis of data using software MINITAB16 and Tukey test were used.

FINDINGS AND ANALYSIS

A. Moisture absorption

The amount of moisture absorption nanocomposites containing nanoparticles are shown in Table 1. Moisture absorption of combined film was CMC + PVA, 03/22 percent. With the addition of 5.0, 1 and 3 percent, nano-clay respectively, 27/1, 99/2 and 073/5 percent reduction was observed in the amount of moisture absorption of the mixed film. (Fakhri *et al*,. 2011) with the addition of 3, 5 and 7%, MMT observed, 8.5, 1/12 and 74/14 percent reduction in moisture on the cellulose films, that results obtained confirms the accuracy of the above. In nanocomposites by increasing the amount of particles, moisture absorption in mixed film also declined. The reasons for this decline can be more coherent structure with high affinity and less free space by adding nanoparticles and also less filler hydrophilic than matrix that reduces permeability of the film moisture inside.

Table 1: Moisture absorption of CMC and its nanocomposites.

Moisture absorption	**films**
A 22/03 ± 0/84	**CMC + PVA**
AB 20/75 ± 2	**CMC+ PVA 0.5%C**
AB 19/04 ± 3/14	**CMC+ PVA 1%C**
B 16/95 ± 2/5	**CMC+ PVA 3%C**

CMC represents carboxymethyl cellulose and PVA represents polyvinyl alcohol, and 0.5, 1, 3% C represents the concentration of Cloisite 30B (modified nanoclays commercial). Different letters in each column indicate statistically significant differences in the level of 95%.

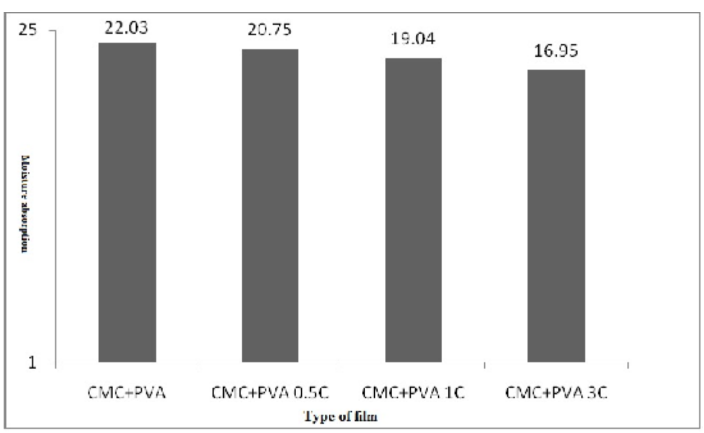

Fig. 1. Effect of C30B (%) on nanocomposite moisture absorption CMC / PVA-nano clay.

B. Determination of water vapor permeability (WVP)

Packing materials should as far as possible, have minimum WVP to prevent the exchange of moisture between environment and food (Park, 2003). Moisture and water vapor is one of the most important reasons for corruption in food-producing reaction is. Water vapor permeability (WVP) is one of the most important features of polymers for food packaging is especially biopolymers. Permeability properties of polymers has direct relation with, hydrophilic or hydrophobic to the nature of their constituents, Process and polymer production, polymer type and amount of additives, the presence of pores and cracks, the curvature of the polymer and the polymer structure (Vaskvnz, 2009). In general, water vapor hydrophilic polymer matrix depends on the diffusion coefficient and solubility coefficient of water molecules in the polymer matrix. Adding nanoparticles to the matrix CMC-PVA, on the one hand by increasing the coherence between the chain and reduce pores (spaces), reduces penetration of water vapor molecules and provides winding pathways for the passage of water molecules. Also, it lowers diffusion rate and thus reduces the WVP. The results of Rahim al in 2007 showed that nanocomposites of modified nanoclay such as C20A and C30B showed significant decrease in permeability to water vapor. While the permeability of the films consists of a non-modified clay and a clay modified with calcium () showed a significant increase. These results can be attributed to the type of clay used and the method of fixing it. Dispersion of hydrophobic Group in The modified nanoclay and hydrophilic groups in non-modified nanoclay natural is a key factor in the reduction or increase of nanocomposites permeability. 3% decreased WVP of nanoclay samples compared to control samples can be attributed to most effectively increase in penetration rate to increase solubility coefficient in permeability and also relatively strong interaction between matrix and filler in clay containing film (Cross, 2008). Cellulose film is a good barrier against oxygen and aromatic compounds and their resistance to water vapor increases with the addition of fat (Kemper, 1984, 1985).

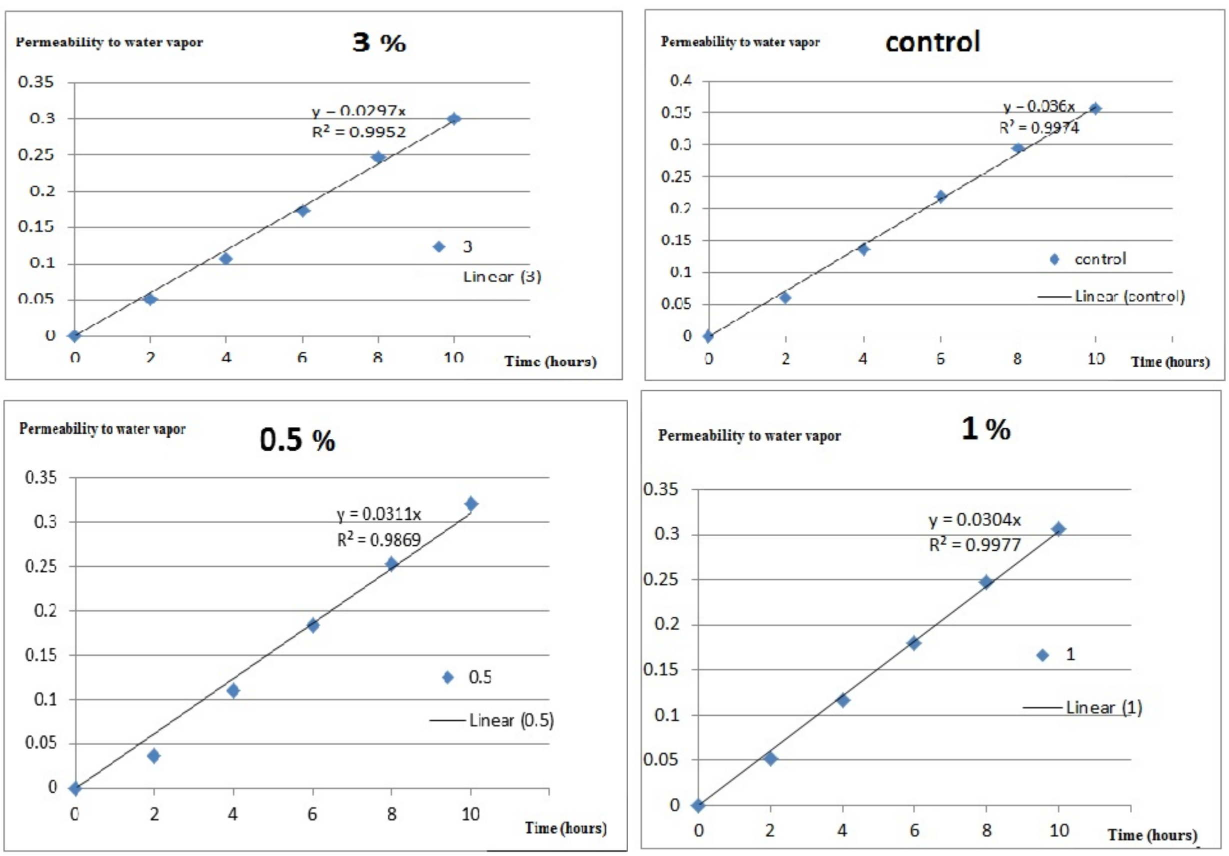

Fig. 2. Effect of C30B (%) on WVP nanocomposite CMC / PVA- nano clay.

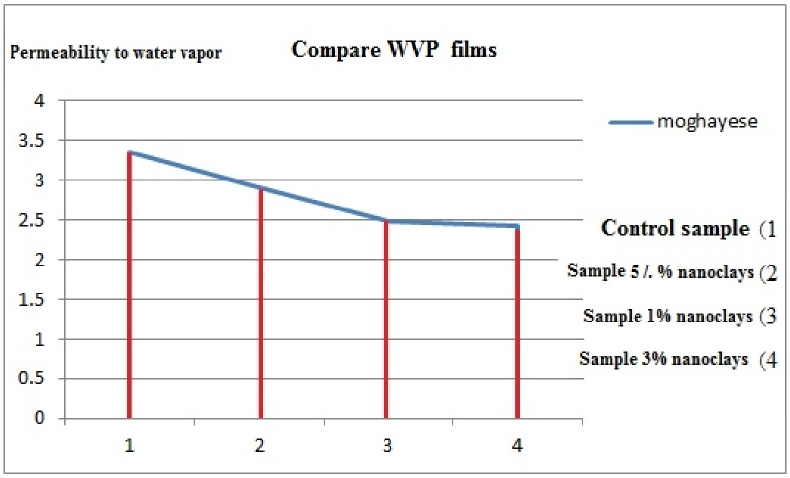

Fig. 3. Comparison of WVP films.

Moisture content: Walnut kernel moisture should not be more than 4% (Standard No. 18, Walnut 1366). Packaging materials should be impervious to oxygen and light. To maintain walnut kernel for more than a few months water vapor permeability of packaging should be minimal (Taj Din *et al.*, 2004). The impact different concentrations of clay were studied in a test and it was found that there was a significant effect of days with humidity over time (P <0.05) and the more time passes, the humidity increases. Various concentrations of moisture affect on humidity (P <0.05) and with increasing concentrations of nanoparticles, humidity decreases. The lowest related to clay samples containing 3% by 63/1 percent. One of the most important reasons for this can be attributed to an effective bulwark against the influence of gases and water vapor by nanoclay attributed (by creating an atmosphere round) so by adding it to biopolymer coating is effectively increases the inhibitory properties of coating (Tang *et al.*, 2009, Adam *et al.*, 2009). Cellulose-based wrapper have been used successfully in Wrapping ignore the new beans and strawberries. Financial brush techniques compared to coating techniques and flooding gives the best results. Financial brush techniques compared to coating techniques and flooding gives the best results and minimizes the loss of moisture in these products (Yrans and Tunisia, 1997).

Table 2: Humidity of (%) Walnut kernel packed with cellulose nanocomposites.

CMC+PVA 3%C	CMC+ PVA 1%C	CMC+PVA 0.5%C	CMC + PVA	Day / Films
C1/19	BC1/19	B1/19	A1/19	.
C1/28 ± 0/05	BC1/34 ± 0/04	B1/38 ± 0/06	A 1/68 ± 0/1	**30**
C1/47 ± 0/05	BC1/54 ± 0/05	B1/61 ± 0/14	A1/96 ± 0/19	**60**
C1/63 ± 0/19	BC1/7 ± 0/2	B1/81 ± 0/21	A2/11 ± 0/11	**90**

CMC represents carboxymethyl cellulose and PVA represents polyvinyl alcohol, and 0.5, 1, 3% C represents the concentration of Cloisite 30B (modified nanoclays commercial). Different letters in each column indicate statistically significant differences in the level of 95%.

The amount of mold and yeast product: Amount of mold and yeast count is given in Table 3. The results of the comparison showed that with the passage of time (90 days) in the treatment the amount of mold count was significantly (P <0.05) reduced however, this decline was greater in the treatment nano contains 3% so that at the end of the period had the least amount of molds and yeasts (log cfu / gr 17/0 ± 4/3), respectively. The results of the effects of oral anti-fungal coatings based on carboxymethyl cellulose containing potassium sorbate on aflatoxin-producing *Aspergillus* species of pistachio of (Ghanbarzadeh *et al*,. 2010) confirms the results. In this study the mold count showed that in the control (uncoated) mold counts were 106 × 02/2 per gram while in subjects covered, no growth of mold was observed.

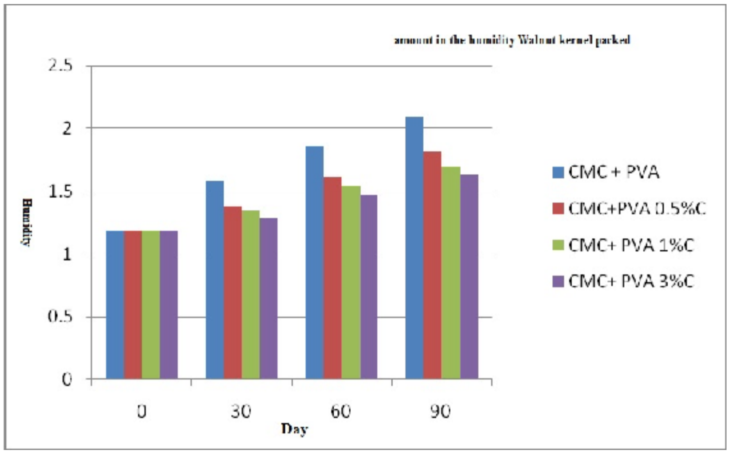

Fig. 4. Changing amount in the humidity Walnut kernel packed with cellulose nanocomposites.

In this study the effect of different concentrations of nano clay and time was investigated. And it turned out that time passage effects on the number of molds and yeasts counts (P <0.05), and over time it will decrease in the product. Different levels affect both its amount and concentration and by increasing concentration of nano, amount of mold and yeast will be reduced (P> 0.05). The two factors of concentration and time together also affect the amount of mold and yeast product.

Table 3: Counting of mold and yeast (cfu / g) Walnut kernel packed with cellulose nanocomposites.

CMC+PVA 3%C	CMC+ PVA 1%C	CMC+PVA 0.5%C	CMC + PVA	Day / Films
^{C}4/46	^{B}4/46	^{AB}4/46	^{A}4/46	.
^{C}3/78 ± 0/28	^{B}4/09 ± 0/17	^{AB}4/12 ± 0/15	^{A} 4/29 ± 0/14	**30**
^{C}3/53 ± 0/2	^{B}3/81 ± 0/13	^{AB}4/04 ± 0/09	^{A}4/15 ± 0/07	**60**
^{C}3/4 ± 0/17	^{B}3/61 ± 0/15	^{AB}3/71 ± 0/23	^{A}4 ± 0/1	**90**

CMC represents carboxymethyl cellulose and PVA represents polyvinyl alcohol, and 0.5, 1, 3% C represents the concentration of Cloisite 30B (modified nanoclays commercial). Different letters in each column indicate statistically significant differences in the level of 95%.

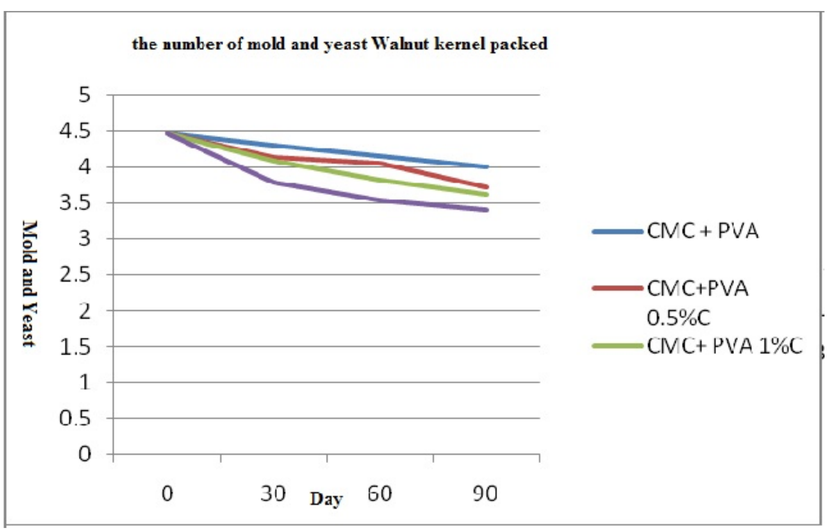

Fig. 5. Changing in the number of mold and yeast Walnut kernel packed with cellulose nanocomposites.

CONCLUSION

In terms of quality, carboxymethyl cellulose material is suitable for food packaging to reduce the influence of gas is water vapor. Today, the biopolymer and its nanocomposites, as well as materials that are combined with softening properties, in order to increase the flexibility of their films, are used in a wide range of food packaging. The aim of food packaging industry is preserving food in the most effective way and with minimal cost so that consumers and manufacturers have the most satisfaction and food remains secure and also to minimize environmental problems. The demand for fresh and safe food production and with minimal process is currently the most important challenges facing today's food packaging industry and always looking for ways to maintain the quality and safety of food packaging. Correct choice of materials and proper packaging cause preserve product quality. The use of modern methods such as the use of nanotechnology for food preservation and survival is important. Of products that have a significant position in the non-oil export development cycle, nuts, including Walnut kernel with appropriate packaging can be up to exchange technology, have a significant share in exports of dried fruits. . According to the results obtained in this study CMC films and nanocomposite carboxymethyl cellulose / polyvinyl alcohol-clay in terms of permeability characteristics can be microbial somewhat competitive with petroleum-derived polymers (such as polyethylene, polypropylene, etc.). And due to problems such as environmental pollution and a lack of adequate resources for the continued use of oil derivatives using biodegradable and renewable resources such biopolymers enough, can change the context of a broad package in the industry so that we can provide high quality products for consumer.

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