# УДОСКОНАЛЕННЯ ВЛАСТИВОСТЕЙ ТОВАРІВ

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## WATERPROOF PAPER PACKAGING MATERIALS: OPTIMIZATION OF HYDROPHOBIC COMPOSITION

*The influence of the three-component system parameters of hydrophobic composition (polyvinyl alcohol, polyamideamineepichlorhydrin, carbamide) on the treated paper properties was investigated. Mathematical models of type "composition - properties" were developed, which allow to optimize the hydrophobic composition ratio components and to obtain paper packaging materials with specified properties (air permeability – 10–30 cm$^3$/min, wet-strength – 30–40% and surface absorption – 10–20 g/m$^2$).*

*Keywords:* paper packaging materials, mathematical modeling, hydrophobic composition, polyvinyl alcohol, polyamideamineepichlorhydrin, carbamide, optimal composition.

***Осыка В., Караваев Т., Комаха В. Влагопрочные бумажные упаковочные материалы: оптимизация гидрофобного состава.*** *Исследовано влияние параметров состава трехкомпонентной системы гидрофобной композиции (поливиниловый спирт, полиамидаминепихлоргидрин, карбамид) на показатели свойств обработанной бумаги. Разработаны математические модели типа "состав –*

*свойства", которые позволили оптимизировать соотношение компонентов гидрофобного состава и получить бумажные упаковочные материалы с заданными свойствами (воздухопроницаемость – 10–30 см$^3$/мин, влагопрочность – 30–40 % и поверхностная впитываемость – 10–20 г/м$^2$).*

*Ключевые слова:* бумажные упаковочные материалы, математическое моделирование, гидрофобный состав, поливиниловый спирт, полиамидаминепихлоргидрин, карбамид, оптимальный состав.

**Background**. The environmental friendliness of packaging materials and their safety is a global problem. The solving of this problem has being attending both leading scientists and practitioners in the packaging industry and international environmental organizations. The development of paper-based packaging materials requires the use of such materials, chemicals and its combinations as would be able to meet all requirements for packaging a wide range of goods. Such requirements determined by the chemical nature of the packaged product, its physical condition, sensitivity to moisture, oxygen, light, the need of total isolation from the environment during storage.

The barrier properties of the paper are usually formed by increasing the material density by using of highly fibrillated cellulose fibers and calendering the paper huckaback made from them, treating by sizing substances or parchment. However, these methods do not provide a high level of wet-strength and waterproof of paper packaging materials (hereinafter – PPM). One of a way to solve this problem is the paper surface treatment with hydrophobic compositions that have the ability to create a protective layer that to prevent the migration of any substance to the packed product from the environment, provide the necessary level of resistance to penetration of moisture, steam, i.e. provide a complex barrier and protective properties.

The scientific research of A. E. Kolosov [1], V. L. Fleisher [2], N. D. Kornienko [3], P. Samyn [4], C. Silvestre [5] are devoted to the problems of the quality forming of paper packaging materials and their properties the study. In particular, these authors propose to use of polymer coatings to obtain packaging materials with the required properties.

In the scientists' research attention is paid to only a few parameters related with the problem of manufacturing PPM with the required properties. At the same time, the complex influence of compositions components on paper processing on the quality of finish products has not been sufficiently investigated.

**Analysis of recent research and publications**. This article is based on the previous study results, which have established an effective fibrous raw material for the production of paper-bases wet-strength and waterproof packaging materials (sulfate unbleached pulp of coniferous and deciduous wood) [6; 7] and optimum grinding rate of cellulose fibers (55-65 degree of Shopper-Rigler (SR) [8]. According to the studies [9] have scientifically substantiated and proved the effectiveness of using epichlorohydrin resins in a composition with polyvinyl alcohol to provide barrier and protective properties of paper packaging materials.

The purpose of the article is to optimize the hydrophobic composition for the production of wet-strength and waterproof paper packaging materials.

**Materials and methods**. Object of study – is the PPM made by treatment of paper-base with hydrophobic compositions.

The subject of the study is the properties of PPM taking into account the change in the hydrophobic composition components ratio.

To give the PPM moisture and water resistance, the paper substrate was subjected to surface treatment with compositions using aqueous solutions of polyamideamineepichlorhydrin (hereinafter – PAAEC), polyvinyl alcohol (hereinafter – PVA) and carbamide.

PAAEC manufactured by Eka Chemicals AB (Sweden) of the EKA WS 325 brand has the following parameters: Brookfield dynamic viscosity – 76.2 MPa·sec, dry matter mass – 19.8%, pH – 3.5.

To ensure better penetration of PAAEC into the structure of the paper, the carbamide of grade B [10] and polyvinyl alcohol of grade 7/18 of high quality [11] produced by PJSC “Severodonetsk AZOT” were used.

The composition was prepared by aqueous solution of the components and mixing them ($\tau \approx$ 20–30 min, T = 30–35 °C). Model formulations with different ratio of the main components were applied to the surface of the paper-base with a mass of 50 g/m$^2$. The paper was dried, stood for 10 days and tested by the methods and regulations adopted in the pulp and paper industry [12–17].

Determination of the solutions optimum composition for providing the paper with wet-strength and waterproof was performed by the multicriteria optimization method using the *STAT-SENS* software.

Mathematical models were obtained using a central composite rotatable plan (CCRP) of experiment with setpoints of output variables in the studied range of values.

Modeling of the process "composition – property" was carried out using of the order 2 model, which has the form [18]:

$$\hat{y} = b_0 + \sum_{i=1}^{k} b_i x_i + \sum_{1 \le i \le j \le k}^{k} b_{ij} x_i x_j + \sum_{i=1}^{k} b_i x_i^2 . \quad (1)$$

To obtain the above model, a plan of a full factorial experiment was used and after implementation, was completed with points in the center of the plan and with the 1.682 star shoulder *(Table 1).*

The significance of the regression coefficients was estimated using the Student's test [19]. To find the rational range of the multicomponent mixture parameters, taking into account a given set of constraints, the multicriteria optimization method was used. The calculations are made with a confidence probability of 0.95. The practical error of experiments did not exceed 5 %, taking into account instrument and accidental errors.

*Table 1*

**Plan of the experiment**

| Experiment sequence number | $x_1$ | $x_2$ | $x_3$ |
|---|---|---|---|
| 1 | –1 | –1 | –1 |
| 2 | 1 | –1 | –1 |
| 3 | –1 | 1 | –1 |
| 4 | 1 | 1 | –1 |
| 5 | –1 | –1 | 1 |
| 6 | 1 | –1 | 1 |
| 7 | –1 | 1 | 1 |
| 8 | 1 | 1 | 1 |
| 9 | 1.6818 | 0 | 0 |
| 10 | –1.6818 | 0 | 0 |
| 11 | 0 | 1.6818 | 0 |
| 12 | 0 | –1.6818 | 0 |
| 13 | 0 | 0 | 1.6818 |
| 14 | 0 | 0 | –1.6818 |
| 15 | 0 | 0 | 0 |

**Results**. The creation of hydrophobic composition to provide wet-strength and waterproof of paper on the basis of PAAEC using PVA and carbamide as functional additives was carried out with the determination of their optimal content in the composition.

Mathematical models have been developed to effectively regulate the properties of the paper-base, enabling the production of packaging materials with specified properties, such as strength in dry and moist condition, surface pickiness and breathability. The most effective for mathematical models constructing is the method of regression analysis, which is to obtain a mathematical description of the packaging material properties dependence on the components ratio in the solution for processing the paper-base.

To limit the number of factors in optimizing process of providing the wet-strength and waterproof of paper-base as the initial variables was selected factors characterizing the ratio of the main components of the hydrophobic composition, wt. %: $x_1$ – PVA content; $x_2$ – content of PAAEC; $x_3$ – carbamide content.

A complete factor experiment was implemented and mathematical models of the form (2) were obtained in order to receive the data array for further optimization of the hydrophobic composition:

$$y = a_0 + a_1x_1 + a_2x_2 + a_3x_3 + a_4x_1x_2 + a_5x_1x_3 + a_6x_2x_3 + a_7x_1^2 + a_8x_2^2 + a_9x_3^2 . \quad (2)$$

To obtain the mathematical dependences of the form $y = f(x_i)$ at $i = 3$ the zero level of the selected factors and their variation interval are set: $x_1 = 2 \pm 1$; $x_2 = 4 \pm 2$; $x_3 = 4 \pm 2$. According to the central composite rotatable experiment plan, 15 model compositions were developed to increase the wet-strength and waterproof of the paper, illustrated by the ratio components examples indicated in the *table 2.*

*Table 2*

**The content of components in the compositions of solutions, wt. %**

| Solution component | Composition number | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | *1* | *2* | *3* | *4* | *5* | *6* | *7* | *8* | *9* | *10* | *11* | *12* | *13* | *14* | *15* |
| PVA | 3 | 1 | 3 | 1 | 3 | 1 | 3 | 1 | 3.6 | 0.4 | 2 | 2 | 2 | 2 | 2 |
| PAAEC | 6 | 6 | 2 | 2 | 6 | 6 | 2 | 2 | 4 | 4 | 7.2 | 0.8 | 4 | 4 | 4 |
| Carbamide | 6 | 6 | 6 | 6 | 2 | 2 | 2 | 2 | 4 | 4 | 4 | 4 | 7.2 | 0.8 | 4 |
| Water | 85 | 87 | 89 | 91 | 89 | 91 | 93 | 95 | 88.4 | 91.6 | 86.8 | 93.2 | 86.8 | 93.2 | 90 |

The efficiency of the process of providing the moisture-strength and waterproof of the paper is determined by the properties obtained after treatment of materials: $y_1$ – air permeability, $cm^3$/min; $y_2$ – destructive force, N; $y_3$ – wet-strength, %; $y_4$ – surface absorption, $g/m^2$.

The obtained results are shown in the *table 3*.

*Table 3*

**The quality of the paper-base treated with a hydrophobic composition at different ratio of the main components**

| Composition number | Component content, wt. % | | | Indicator of the properties | | | |
|---|---|---|---|---|---|---|---|
| | PVA | PAAEC | Carbamide | Air permeability, $cm^3$/min | Destructive force, N | Wet-strength, % | Surface absorption, $g/m^2$ |
| 1 | 3 | 6 | 6 | 19 | 78 | 40 | 15 |
| 2 | 1 | 6 | 6 | 28 | 55 | 35 | 21 |
| 3 | 3 | 2 | 6 | 22 | 71 | 25 | 26 |
| 4 | 1 | 2 | 6 | 39 | 53 | 19 | 34 |
| 5 | 3 | 6 | 2 | 20 | 75 | 32 | 15 |
| 6 | 1 | 6 | 2 | 29 | 51 | 20 | 17 |
| 7 | 3 | 2 | 2 | 20 | 70 | 30 | 24 |
| 8 | 1 | 2 | 2 | 38 | 50 | 17 | 28 |
| 9 | 3.6 | 4 | 4 | 14 | 86 | 36 | 17 |
| 10 | 0.4 | 4 | 4 | 38 | 48 | 26 | 23 |
| 11 | 2 | 7.2 | 4 | 24 | 63 | 39 | 14 |
| 12 | 2 | 0.8 | 4 | 34 | 58 | 21 | 31 |
| 13 | 2 | 4 | 7,2 | 29 | 65 | 27 | 21 |
| 14 | 2 | 4 | 0,8 | 28 | 60 | 20 | 19 |
| 15 | 2 | 4 | 4 | 36 | 62 | 31 | 16 |

Adding of polyamideamineepichlorhydrin resin to the composition with carbamide contributes not only to the reduction of permeability, but also to increase the ductility and mechanical strength in the dry and humidity states.

The obtained results of studies and tests of treated paper samples confirm that the use of PVA in model solutions, even with low content (1–2 wt. %) in combination with PAAEC contributes to the creation of a more tight microporous structure that confirmed by surface absorption indices of the treated paper.

The analysis of the strength indices of the test samples treated with different variants of the model solutions shows that the content of carbamide does not significantly affect on the paper wet-strength. The main increase in mechanical strength, especially in the wet state, is ensured by the increase of the PAAEC content in the model solution, which reach the optimal properties in the range of 4–6 %.

The use of the solution with PVA content of more than 3 % is unreasonably, since the high viscosity of the composition leads to its poor penetration into the cellulose fibers, requires an increase of the time to leak the paper, causing the coating to stick to the drying cylinders during drying. As a consequence, it causes to the paper quality deteriorate by some indicators, such as wet-strength, and damage its structure.

According to the obtained array of experimental data, mathematical models of components influence indices on the study system properties (3–6) were created, according to which the packing material were tested:

a) by the air permeability index ($y_1$):

$$y_1 = +80.089 - 9.8363x_1 - 1.9156x_2 + 0.19637x_3 + 0.125x_1x_2 + \\ +1.125x_1x_3 - 0.625x_2x_3 - 6.6584x_1^2 - 0.5978x_2^2 - 2.7746x_3^2; \quad (3)$$

b) by the destructive force index ($y_2$):

$$y_2 = +39.103 + 8.904x_1 + 2.7141x_2 + 1.4212x_3 + 1.125x_1x_2 - 0.375x_1x_3 + \\ +0.375x_2x_3 + 1.6246x_1^2 - 0.67348x_2^2 + 0.033621x_3^2; \quad (4)$$

c) by the wet-strength index ($y_3$):

$$y_3 = +2.143 + 3.8675x_1 + 4.8527x_2 + 2.3265x_3 - 0.25x_1x_2 - \\ -1.75x_1x_3 + 3.25x_2x_3 - 0.19774x_1^2 - 0.55129x_2^2 - 2.8494x_3^2; \quad (5)$$

d) by the surface absorption index ($y_4$):

$$y_4 = +25.737 - 2.2033x_1 - 7.3153x_2 + 1.125x_3 + 0.5x_1x_2 - \\ -1x_1x_3 - 0.5x_2x_3 + 1.7789x_1^2 + 0.6627x_2^2 + 1.7789x_3^2. \quad (6)$$

The study of mathematical descriptions was conducted to determine and establish patterns of an object behavior in conditions that were not considered in the experiment. The developed models with high precision can be used to calculate the values of the solution-impregnated packing material with any concentration of components. Such models make it possible to predict changes in the relevant indicators in situations and conditions that are not reflected experimentally. When calculating the optimal composition of the solution it is necessary to take into account that all the components of the composition interact therefore to change the value of a certain index by changing the concentration of only one component is impossible. For this reason, the determination of the solution optimum composition for waterproof providing of the paper packaging material was carried out by the multicriterion optimization method that allow to obtain the value of the factors that characterize the optimal composition, in order to provide the specified barrier and protective properties of the packaging material for moisture-content products.

The main indices for calculating the optimal composition are shown in the *table 4*. The upper values of these indices and the range of the wet-strength and air permeability indices have been established taking into account the results of the experimental studies and are maximum acceptable. They ensure compliance with the lower levels of the key indicators.

*Table 4*

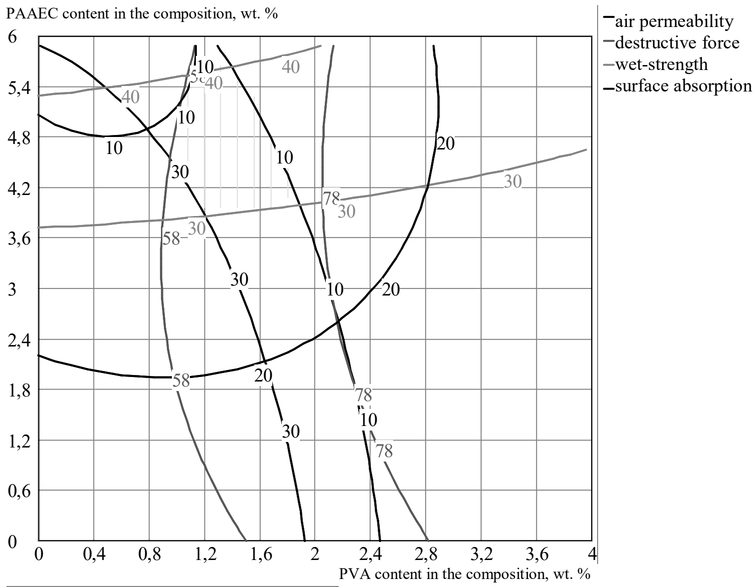
**Optimization parameters of hydrophobic composition for providing wet-strength and waterproof to paper packaging materials**

| Criteria and calculation of optimization | Air permeability, м$^3$/min | Destructive force, N | Wet-strength, % | Surface absorption, g/m$^2$ |
|---|---|---|---|---|
| The minimum value | 10 | 58 | 30 | 10 |
| The maximum value | 30 | 78 | 40 | 20 |

The parameters and results of finding the components optimal ratio of the hydrophobic composition are shown on the *figure*.

Therefore, treatment of the paper-base with hydrophobic composition containing, wt. %: 1–2 PVA, 4–6 PAAEC, 4–6 carbamide and water as a solvent, gives the paper water resistance, wet-strength and also increases the level of barriers properties and reduces its surface absorption.

According to the regression analysis results, mathematical models of the treated paper change depending on the solution components content were created.




Compromise optimal composition area
to provide surface absorption and wet-strength of the paper

*Source*: Created by the authors.

These models application allows to predict the value of a particular index, which made it possible to optimize the hydrophobic composition to provide the paper with increased water resistance, wet-strength and other strength and barrier properties.

**Conclusion**. The main increase of mechanical strength of the paper, especially in wet state, is ensured with the PAAEC increase in the range of 4–6 wt. % in the model solution content.

The PVA usage in model solutions in the range of 1–2 wt. % together with PAAEC contribute to the creation of a more closed microporous structure, as evidenced by the surface absorption index of the treated samples. The use of the solution with PVA content of more than 3 wt. % is unreasonably, since the high viscosity of the composition leads to its poor penetration into the cellulose fibers, requires an increase of the time to leak the paper, causing the coating to stick to the drying cylinders.

The optimal components range of the hydrophobic composition was determined, wt. %: PAAEC – 4–6, PVA – 1–2, carbamide – 4–6, which gives to the PPM the specified barrier and strength properties (air permeability – 10–30 $cm^3$/min , wet-strength – 30–40 %, surface absorption –10–20 $g/m^2$).

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***Осика В., Караваєв Т., Комаха В. Вологоміцні паперові пакувальні матеріали: оптимізація гідрофобного складу.***

**Постановка проблеми.** Для надання паперу заданих показників бар'єрних і міцнісних властивостей папір-основу піддавали поверхневому обробленню гідрофобними композиціями. Для ефективного регулювання властивостей паперу-основи розроблено математичні моделі, що дають змогу виготовляти пакувальні матеріали із такими заданими властивостями, як міцність у сухому та вологому стані, поверхнева вбирність, повітропроникність.

*Мета статті* – оптимізація гідрофобного складу для отримання вологоміцних водонепроникних паперових пакувальних матеріалів.

**Матеріали та методи.** Папір піддавали поверхневому обробленню композиціями з використанням водних розчинів поліамідамінепіхлоргідрину, полівінілового спирту та карбаміду.

Методом регресійного аналізу експериментальних даних з використанням центрального композиційного рототабельного плану отримано математичні описи залежності властивостей паперу від вмісту компонентів гідрофобного складу. Математична обробка результатів експерименту здійснювалась із використанням програмного забезпечення STAT-SENS.

**Результати дослідження**. Основний приріст механічної міцності, особливо у вологому стані, забезпечується завдяки підвищенню вмісту у розчині поліамідамінепіхлоргідрину, який проявляє оптимальні властивості в діапазоні 4–6 %. Використання полівінілового спирту у розчинах навіть за незначного вмісту (1–2 мас.%) сумісно з поліамідамінепіхлоргідрином сприяє створенню більш зімкнутої мікропористої структури, про що свідчать показники поверхневої вбирності оброблених зразків.

**Висновки.** Визначено оптимальний діапазон вмісту компонентів гідрофобного складу, %: ПААЕХ – 4–6, ПВС – 1–2, карбаміду – 4–6, що надає ППМ заданих показників бар'єрних і міцнісних властивостей: повітропроникність – 10–30 $см^3$/хв, вологоміцність – 30–40 %, поверхневу вбирність – 10–20 г/$м^2$.

*Ключові слова:* паперові пакувальні матеріали, математичне моделювання, гідрофобний склад, полівініловий спирт, поліамідамінепіхлоргідрин, карбамід, оптимальний склад.