

УДК 621.798.2:676.24

DOI: 10.31617/2.2023(45)08

Viktor OSYKA

Doctor of Technical Sciences,
Dean of the Faculty of Trade and Marketing,
Professor at the Department of Commodity
Science, Safety and Quality Management
State University of Trade and Economics
19, Kyoto St., Kyiv, 02156, Ukraine
v.osyka@knute.edu.ua

Віктор ОСИКА

д. т. н., професор,
декан факультету торгівлі та маркетингу,
Державний торговельно-економічний
університет

вул. Кіото, 19, м. Київ, 02156, Україна
ORCID: 0000-0002-5081-7727

Oliha KOMAKHA

PhD (Technical Sciences),
Associate Professor, Associate Professor
at the Department of Commodity Science
and Customs Affairs
State University of Trade and Economics
19, Kyoto St., Kyiv, 02156, Ukraine
o.komakha@knute.edu.ua

Ольга КОМАХА

к. т. н., доцент, доцент кафедри
товарознавства та митної справи
Державний торговельно-економічний
університет

вул. Кіото, 19, м. Київ, 02156, Україна
ORCID: 0000-0003-0312-890X

Volodymyr KOMAKHA

PhD (Technical Sciences),
Associate Professor, Associate Professor
at the Department of Commodity Science
and Customs Affairs
State University of Trade and Economics
19, Kyoto St., Kyiv, 02156, Ukraine
v.komakha@knute.edu.ua

Володимир КОМАХА

к. т. н., доцент, доцент кафедри
товарознавства та митної справи
Державний торговельно-економічний
університет

вул. Кіото, 19, м. Київ, 02156, Україна
ORCID: 0000-0001-6498-9047

**PAPER PACKAGING MATERIALS:
MODELING AND OPTIMIZATION
OF HYDROOLEOPHOBIC
PROPERTIES**

**ПАПЕРОВІ ПАКУВАЛЬНІ
МАТЕРІАЛИ: МОДЕЛЮВАННЯ
ТА ОПТИМІЗАЦІЯ
ГІДРООЛЕОФОБНИХ ВЛАСТИВОСТЕЙ**

Introduction. A high level of barrier and protective properties, resistance to moisture penetration (water, steam) and air resistance are the main requirements for packaging paper for food products.

Problem. Polyvinyl alcohol-based polymer coatings are widely used for packaging paper; however, such coatings tend to have poor water resistance due to the hydrophilic and water-soluble nature of polyvinyl alcohol. Polyamide-epichlorohydrin resins are used to provide moisture resistance of the paper, and glycerol is used for the elasticity of the coating. At the same time, the complex effect of these components in the mixture for surface treatment of paper on the quality of the products has not been sufficiently investigated.

Вступ. Високий рівень бар'єрних і захисних властивостей, стійкості до проникнення вологи (води, пари) і повітря є основною вимогою до пакувального паперу для харчових продуктів.

Проблема. Полімерні покриття на основі полівінілового спирту широко використовуються для виготовлення пакувального паперу, однак вони мають низьку водостійкість через гідрофільну та водорозчинну природу полівінілового спирту. Для надання вологоміцності паперу використовують поліамідамінепіхлоргідринові смоли, а для еластичності покриття – гліцерин. При цьому комплексний вплив зазначених компонентів в суміші для поверхневого оброблення паперу на якість виробів недостатньо досліджений.

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Contribution of the authors: Osyka V. – 60 %, Komakha O. – 20 %, Komakha V. – 20 %.

The authors received no direct funding for this study.

Osyka V., Komakha O., Komakha V. Paper packaging materials: modeling and optimization of hydro-oleophobic properties. *Mizhnarodnyj naukovo-praktychnyj zhurnal "Tovary i rynky"*. 2023. № 1 (45). S. 89-99. [https://doi.org/10.31617/2.2023\(45\)08](https://doi.org/10.31617/2.2023(45)08)

Methods. Compositions based on aqueous solutions of polyvinyl alcohol brand 7/18 of the highest grade, polyamideamineepichlorhydrin EKA WS 325 and glycerol brand PK-94 were used to obtain a moisture-resistant, waterproof and fat-proof packaging material. Polyacrylamide in the amount of 0.25 wt. % was used as a functional additive, a viscosity regulator of the composition, and water was used as a solvent. Model compositions with different ratios of the main components in accordance with the central composite rotatable plan of the experiment were applied to the tests according to the methods adopted in the pulp and paper industry. STAT-SENS software was used for mathematical processing of the experimental results. A multi-criteria optimization method was used to find the optimal range of parameters of the hydrooleophobic composition.

Results. The 15 model compositions have been developed. The influencing factors were the content of the polyvinyl alcohol, polyamideamineepichlorhydrin, glycerol. The quality indicators of the treated paper-base were selected as the response functions of mathematical models: oil permeability, air permeability, destructive force, moisture resistance, surface absorption.

Conclusions. The developed composition is optimal and makes it possible not to exceed its consumption during application to fibrous material, in particular paper. The composition penetrates the thickness of the paper to an optimal depth evenly over the entire surface of the paper, which makes it possible to provide the paper with uniform barrier properties, mechanical strength and wet-strength along the plane of the canvas. In addition, glycerol gives elasticity to the resulting coating and prevents it from cracking during repeated bending.

Keywords: paper packaging materials, polyvinyl alcohol, polyamideamineepichlorhydrin, wet-strength, oil resistance, water resistance, properties modeling, multi-criteria optimization.

Методи. Використано композиції на основі водних розчинів полівінілового спирту марки 7/18 вищого гатунку, поліамідамінепіхлоргідрину ЕКА WS 325 та гліцерину марки ПК-94. Як функціональну добавку, регулятор в'язкості складу, застосовано поліакриламід у кількості 0.25 мас. %, а як розчинник – воду. Досліджено модельні склади з різними співвідношеннями основних компонентів за прийнятими в целюлозно-паперовій промисловості методами. Для математичної обробки результатів використано STAT-SENS. Задля пошуку оптимальної області параметрів гідроолеофобного складу застосовано метод багатокритеріальної оптимізації.

Результати дослідження. Розроблено 15 модельних складів. Факторами впливу визначено вміст у композиції компонентів суміші: ПВС, ПААЕХ, гліцерину. Функціями відгуку математичних моделей обрано показники якості обробленого паперу-основи: жиропроникність, повітропроникність, руйнівне зусилля, вологоміцність, поверхнева вбирність.

Висновки. Розроблений склад має оптимальну композицію, що дає змогу не перевищувати його витрати під час нанесення на волокнистий матеріал, зокрема папір. Склад проникає в товщу паперу на оптимальну глибину рівномірно по всій площі паперу, що уможливорює надання паперу рівномірних бар'єрних властивостей, механічної міцності та вологоміцності по площині полотна. Крім того, гліцерин додає еластичності отриманому покриттю та запобігає його розтріскуванню під час багаторазових перегинів.

Ключові слова: паперові пакувальні матеріали, полівініловий спирт, поліамідамінепіхлоргідрин, вологоміцність, жиронепроникність, водонепроникність, моделювання властивостей, багатокритеріальна оптимізація.

Introduction. The use of paper-based packaging materials is explained by their ecological safety and minimal impact on the environment, their ability to provide the required level of barrier properties, and the possibility of reusing their waste as a valuable raw material for the production of new products. The development of the paper packaging production requires the use of new materials and their compositions to ensure the preservation of the consumer properties of packaged products from the effects of the environment [1–4].

During the storage of food products, complex biochemical processes of interaction between products, packaging and the natural environment occur under the influence of various factors (moisture, steam, gas). A high

level of barrier and protective properties, resistance to moisture penetration (water, steam) and air resistance are the main requirements for food packaging paper [5].

Problem. The results of numerous studies show that the barrier properties of paper are formed, usually, by increasing the density of the material by using highly fibrillated cellulose fibers and calendering the paper fabric made from them, by treating it with sizing agents or parchment [6; 7]. However, these methods do not provide a high level of wet-strength, oil and water resistance of paper packaging materials [8–9].

To improve the barrier properties of capillary-porous paper, one of the effective methods is to close the pores on the paper surface and thus block the diffusion path of liquid/gas molecules through the paper. The general approach of creating paper packaging materials is to apply polymers with good film-forming characteristics to the paper substrate and thus form a continuous and impermeable film [10].

Polvinyl alcohol (hereinafter – PVA) based polymer coatings are widely used for the production of packaging paper due to its film-forming ability, good barrier and mechanical properties [11; 12; 13]. However, such coatings, as a rule, have low water resistance due to the hydrophilic and water-soluble nature of PVA [14].

Analysis of recent research and publications. In order to form the barrier properties of paper, polymer coatings based on PVA are used due to its ability to form impermeable films. Numerous researches show that the use of PVA led to an increase in the resistance of packaging paper to water vapor (from 533 to 1.3 g/m²/24 h) and water resistance (wetting angle $\geq 100^\circ$) [12], mechanical strength of paper materials in the dry state [13; 14]. However, the disadvantage of paper treated with PVA, which makes it difficult to use it for wet and fat-containing food products, is the low-level mechanical strength of paper in the wet state and the low elasticity of the coating. Polyamide-epichlorohydrin (hereinafter – PAE) resins [15; 16] are used to provide wet-strength to paper, and glycerol is used for elasticity of the coating. At the same time, the complex effect of these components in the mixture for surface treatment of paper on the quality of the products has not been sufficiently investigated. In the research of scientists on the problem of manufacturing paper packaging materials with specified properties, attention is paid only to certain parameters.

This article is based on the results of previous studies [5; 8; 17], where it was determined that the most effective fibrous raw material for the production of base paper is sulfate-bleached and unbleached cellulose from coniferous wood species at the optimal degree of grinding of cellulose fibers 65-75° SR. Also, the effectiveness of using PVA in a composition with PAE and glycerol for the formation of barrier and protective properties of paper has been tested and proven [18].

In order to achieve a high level of barrier properties, elasticity, mechanical strength in dry and wet conditions, it is proposed to treat the

surface of the base paper with a hydrooleophobic composition based on PVA, PAE and glycerol.

The aim of the research is to develop mathematical models of the dependence of the packaging material properties on the content of the main components of the composition for processing the base paper and the optimization of the hydrooleophobic composition to obtain paper packaging materials with the specified properties.

Methods. To give packaging paper materials wet-strength, oil and water resistance, the surface of the base paper was treated with compositions using aqueous solutions of polyvinyl alcohol (PVA), polyamideamineepi-chlorohydrin (PAE) and glycerol.

We used PAE produced by *Eka Chemicals AB* (Sweden) brand *EKA WS 325* (dynamic viscosity according to Brookfield – 76.2 mPa·s, mass fraction of dry substances – 19.8 %; pH 3.5), PVA produced by *PrJSC "Severodonetske combination of AZOT"* grade 7/18 of the highest grade (dynamic viscosity of a 4 % solution – 28.0 Pa·s·10³; mass fraction of acetate groups – no more than 19.8 %; pH of a 4 % aqueous solution – 4.5).

At low humidity, PVA coatings become brittle, and at high humidity, they may lose their tensile strength. A number of functional additives were used to stabilize the properties of PVA: PK-94 glycerol (saponification coefficient – 0.7 mg/g KOH, density at 20 °C – 1.25 g/cm³), which is used in the pharmacological, food and cosmetic industries and polyacrylamide manufactured by *Kemira Oyj* (Finland) *Fennopol K 3450* (dynamic viscosity of a 5 % solution – 41.0 Pa·s·10³; pH of a 5 % aqueous solution – 6).

In order to ensure the maximum solubility of all components of the composition, a polar substance (water) was chosen as a solvent. The composition was prepared by mixing the components ($\tau \approx 20\text{--}30$ min., $T = 30\text{--}35$ °C). The scheme for preparing a hydrooleophobic composition for paper processing is shown in *Figure 1*.

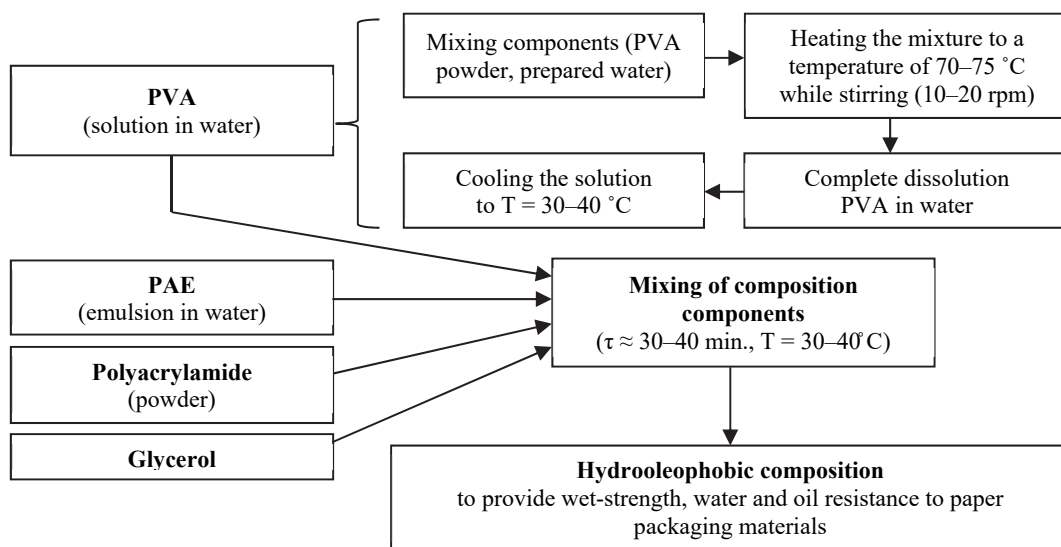


Figure 1. Scheme of preparation of a hydrooleophobic composition to provide wet-strength, water and oil resistance to paper packaging materials

Model compositions were applied to the surface of paper weighing 45 g/m², the samples were dried, conditioned for 10 days and tested according to the methods adopted in the pulp and paper industry [19–23].

Mathematical models of the 2nd order of the type "composition – property" (1) were obtained by processing an array of experimental data using the STAT-SENS software [24].

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

The central composite rotatable plan of the full factorial experiment was used, completed with points in the center of the plan and a star arm of 1.682 (Table 1).

Table 1

Experiment plan

| Serial number of the experiment | 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 |

The method of multi-criteria optimization was used to find a rational range of parameters of a multi-component mixture for paper processing, taking into account a given set of limitations of the quality indicators of the packaging material. Calculations were carried out with a confidence probability of 0.95.

Results. Modeling of a multicomponent hydrooleophobic composition based on PVA, PAE and glycerol to give paper packaging materials wet-strength, water and grease resistance was carried out using the implementation of a central composite rotatable experimental plan. When planning an experiment on the study of changes in the quality indicators of packaging paper, the indicators of the content of the main components of the hydrooleophobic composition were chosen as the initial variables, wt. %, namely x_1 – PVA, x_2 – PAE, x_3 – glycerol. The quality indicators of the treated paper-base were selected as the response functions of the mathematical models: Y_1 – oil permeability, mg; Y_2 – air permeability, cm³/min; Y_3 – destructive force, N; Y_4 – wet-strength, %; Y_5 – surface absorption, g/m².

The study of the influence of the components of the solution for processing the paper-base and the technological parameters of their application made it possible to establish the area of the experiment to obtain the dependences of the form $Y = f(x_i)$ at $i = 3$. At the same time, the center of the orthogonal rotatable plan is located at the points $x_1 = 10, x_2 = 3, x_3 = 2$, with variation intervals of $\pm 4, \pm 1, \pm 1$, respectively.

According to the central composite rotatable plan, 15 model compositions were developed to increase the wet-strength, water and oil resistance of paper, which are illustrated by examples (Table 2).

Table 2

**Compositions of solutions to provide wet-strength,
water and oil resistance to the paper-base**

| The name of the solution component | Content of solution components, wt. % (depending on the version of the composition) | | | | | | | | | | | | | | |
|------------------------------------|--|----|----|----|----|----|----|----|-------|-------|-------|-------|-------|-------|----|
| | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 14 | 15 |
| PVA | 14 | 6 | 14 | 6 | 14 | 6 | 14 | 6 | 16.73 | 3.27 | 10 | 10 | 10 | 10 | 10 |
| PAE | 4 | 4 | 2 | 2 | 4 | 4 | 2 | 2 | 3 | 3 | 4.69 | 1.32 | 3 | 3 | 3 |
| Glycerol | 3 | 3 | 3 | 3 | 1 | 1 | 1 | 1 | 2 | 2 | 2 | 2 | 3.69 | 0.32 | 2 |
| Water | 79 | 87 | 81 | 89 | 81 | 89 | 83 | 91 | 78.7 | 91.73 | 83.31 | 86.68 | 83.31 | 86.68 | 85 |

The model compositions were applied to the surface of the paper-base with a weight of 45 g/m^2 , the obtained samples were dried, conditioned and tested according to the standards adopted in the pulp and paper industry.

The obtained results are shown in the Table 3.

Table 3

Quality indicators of paper-base treated with model hydrooleophobic compounds

| Composition number | Content of the component, wt. % | | | Property indicators | | | | |
|--------------------|---------------------------------|------|----------|----------------------|--|----------------------|-----------------|------------------------------------|
| | PVA | PAE | Glycerol | Oil permeability, mg | Air permeability, cm^3/min | Destructive force, N | Wet-strength, % | Surface absorption, g/m^2 |
| 1 | 14 | 4 | 3 | 0.31 | 2.9 | 81 | 44.6 | 7.9 |
| 2 | 6 | 4 | 3 | 9.1 | 11.1 | 56 | 17.1 | 16.2 |
| 3 | 14 | 2 | 3 | 0.36 | 2.2 | 76 | 38.1 | 7.1 |
| 4 | 6 | 2 | 3 | 11.2 | 10.1 | 53 | 12 | 17.3 |
| 5 | 14 | 4 | 1 | 0.38 | 2.8 | 79 | 43.2 | 8.2 |
| 6 | 6 | 4 | 1 | 12.2 | 10.2 | 52 | 16.2 | 16.9 |
| 7 | 14 | 2 | 1 | 0.41 | 2.8 | 74 | 37.8 | 9.1 |
| 8 | 6 | 2 | 1 | 12.9 | 10.1 | 49 | 11.1 | 18.4 |
| 9 | 16.73 | 3 | 2 | 0.2 | 3.1 | 78 | 43.9 | 6.4 |
| 10 | 3.27 | 3 | 2 | 19.1 | 19.9 | 49 | 10.2 | 38 |
| 11 | 10 | 4.69 | 2 | 2.2 | 4.2 | 72 | 44.8 | 10.2 |
| 12 | 10 | 1.32 | 2 | 2.6 | 4.8 | 64 | 32.2 | 12.2 |
| 13 | 10 | 3 | 3.69 | 1.8 | 4.2 | 69 | 38 | 10.7 |
| 14 | 10 | 3 | 0.32 | 2.4 | 4.8 | 66 | 33.3 | 11.8 |
| 15 | 10 | 3 | 2 | 2.3 | 4.6 | 68 | 36.5 | 11.7 |

To obtain mathematical models, a regression analysis of the obtained array of experimental data was carried out. As a result, we obtained equations describing the influence of the components of the hydrooleophobic composition on the properties of the studied system (2–6):

a) mathematical model based on the oil permeability indicator (Y_1):

$$Y_1 = + 35.371 - 5.6087x_1 - 1.8475x_2 - 2.1391x_3 + 0.0849x_1x_2 + 0.126x_1x_3 - 0.1375x_2x_3 + 0.1887x_1^2 + 0.3985x_2^2 + 0.2936x_3^2 \quad (2)$$

b) mathematical model based on the air permeability indicator (Y_2):

$$Y_2 = + 27.853 - 3.8547x_1 + 0.7074x_2 + 0.4074x_3 - 0.0125x_1x_2 - 0.0437x_1x_3 + 0.1998x_2x_3 + 0.1449x_1^2 - 0.1539x_2^2 - 0.1534x_3^2 \quad (3)$$

c) mathematical model based on the indicator of destructive force in the dry state (Y_3):

$$Y_3 = + 16.463 + 4.6844x_1 + 2.8316x_2 + 4.4889x_3 + 0.1253x_1x_2 - 0.1247x_1x_3 + 0.0011x_2x_3 - 0.1194x_1^2 - 0.3217x_2^2 - 0.4987x_3^2 \quad (4)$$

d) mathematical model based on the wet-strength indicator (Y_4):

$$Y_4 = - 47.362 + 7.997x_1 + 6.5979x_2 + 8.9498x_3 + 0.0533x_1x_2 - 0.0302x_1x_3 + 0.1382x_2x_3 - 0.2875x_1^2 - 0.9723x_2^2 - 0.8723x_3^2 \quad (5)$$

e) mathematical model based on the indicator of surface absorption (Y_5):

$$Y_5 = + 42.935 - 5.6675x_1 + 2.1912x_2 + 1.7383x_3 + 0.1181x_1x_2 - 0.0156x_1x_3 + 0.2625x_2x_3 + 0.1911x_1^2 - 0.8228x_2^2 - 0.7691x_3^2 \quad (6)$$

All three factors that characterize the quantitative ratio of the components of the hydrooleophobic composition affect the oil permeability of the packaging material. The most significant influence on the oil permeability of the packaging material is the content of PVA and glycerin in the composition of which the processing was carried out.

The use of a composition containing PVA less than 6 wt. % and glycerol less than 0.5 wt. %, for the purpose of making the paper greaseproof is impractical, because the oil resistance of the fibrous material does not reach the required level. At the same time, for this purpose, it is impractical to use a grease proofing composition containing more than 10.0 wt. % PVA and more than 3.0 wt. % glycerol, as the cost of paper production increases, and the level of oil resistance increases to a small extent.

It should be noted that taking into account the analysis of literary sources, patent and technical information and based on previous studies on the influence of components and their combinations on the properties of the paper-base, the design of the experimental plan was carried out in the area that is close to the desired optimum.

According to the developed models, it is possible to calculate the values of the indicators of the packaging material obtained by processing with a hydrooleophobic solution with different concentrations of components with sufficiently high accuracy.

The obtained mathematical models made it possible to begin the implementation of the next stage of research, the task of which is to develop a rational ratio of the components of the hydrooleophobic composition.

The content of the components in the composition for paper processing was determined by the method of multi-criteria optimization in order to provide the specified barrier and protective properties of the moisture-resistant, water- and greaseproof packaging material. The main indicators used to calculate the optimal composition of the hydrooleophobic composition are: grease resistance, which should not be higher than 3 mg; air permeability – no higher than 8 cm³/min.; destructive force in the dry state – in the range of 60–70 N, wet-strength – not lower than 25 % and surface water absorption – not more than 8 g/m².

The parameters and results of the search for the optimal ratio of components of the hydrooleophobic composition are given in the *Table 4* and *Figure 2*.

Table 4

Optimization parameters of the hydrooleophobic composition to provide wet-strength and waterproofing to paper packaging materials

| Optimization criteria and calculation | Oil permeability, mg | Air permeability, cm ³ /min | Destructive force, N | Wet-strength, % | Surface absorption, g/m ² |
|---------------------------------------|----------------------|--|----------------------|-----------------|--------------------------------------|
| Minimum value | 0.5 | 5 | 60 | 25 | 5 |
| Maximum value | 3 | 8 | 70 | 30 | 8 |

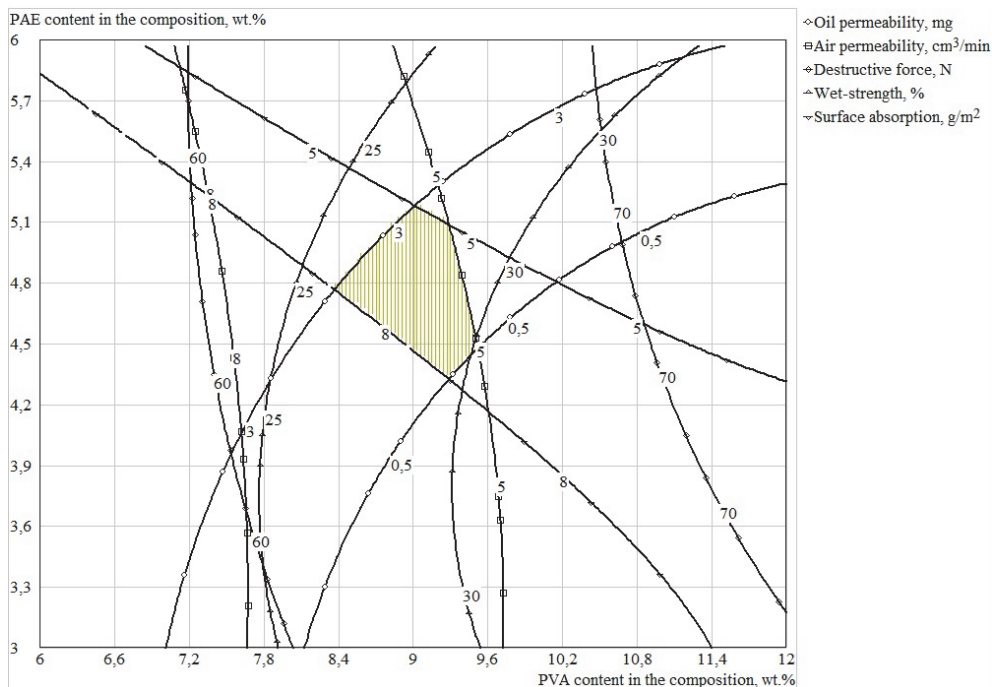


Figure 2. Compromise optimal range of the composition to provide wet-strength, water and oil resistance of the paper

The optimal ratio of the components of the hydrooleophobic composition to provide greaseproofness and wet-strength to paper packaging materials contains the following ratio (wt. %) of the main components: PVA – 8–9, PAE – 4–5; glycerol – 1.0–1.5. Polyacrylamide in the amount of 0.25 wt. % was used as a functional additive, a viscosity regulator of the composition, and water was used as a solvent.

Conclusions. The developed composition is optimal, which makes it possible not to exceed its consumption during application to fibrous material, in particular paper. The composition penetrates the thickness of the paper to the optimal depth evenly over the entire paper area. This circumstance makes it possible to provide the paper with uniform oil resistance along the plane of the canvas. In addition, glycerin gives elasticity to the resulting coating and prevents it from cracking during repeated bending. After treating the paper-base with a hydrooleophobic composition, its strength index during double bending increases from 45 to 65 N, i. e. almost by 1.5–2 times. This property of the composition allows not to lose greaseproofness in places of bends and to achieve uniformity of greaseproofness over the entire area of the paper web during its operation. Due to this, a complex of operational properties is ensured: a dense and closed structure; high oil resistance; increasing the mechanical strength of paper, its elasticity, plasticity and flexibility.

Conflict of interest. The authors certify that they have no financial or non-financial interest in the subject matter or materials discussed in this manuscript; the authors have no association with state bodies, any organizations or commercial entities having a financial interest in or financial conflict with the subject matter or research presented in the manuscript. The authors are working for the institution that publishes this journal, which may cause potential conflict or suspicion of bias and therefore the final decision to publish this article (including the reviewers and editors) is made by the members of the Editorial Board who are not the employees of this institution.

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Received at the editorial office 27.02.2023.

Accepted for printing 03.03.2023.

Published online 23.03.2023.