

## Performance Evaluation of Coating Materials Containing Perlite-Volcanic Lava Stone-Carrot Pulp Ternary System

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**Summary:** Cellulose in the fibrous structure, which is the main structural component in the cell wall of plants, was formed by the combination of three thousand or more glucose molecules and was a natural polymer synthesized by many living microorganisms. In this study, it was aimed to observe the performance of carrot pulp physically added to water-based coating to increase the viscosity of the coatings. In addition, volcanic lava stone (red pumice stone) and perlite stone were added to the coating to improve its properties that heat transfer, adhesion, hydrophobicity, corrosion resistance. The effect of cellulose-based material on the viscosity of the coating was determined using the Design Expert Optimization Method. In this experimental design method, the experimental set was created to be "carrot pulp, volcanic lava stone, and perlite stone" and additives was added as 0-6% by weight of the coating. By using this method, the most suitable process parameters were determined, and the effects of the additives added to the coating were examined, comparatively. As a result, it was found that cellulose derived organic additives and other additives improved the various properties of coating and could be evaluated for industrial coatings.

**Keywords:** Carrot pulp, coating, volcanic lava stone, cellulose, perlite

### Introduction

Cellulose is a natural polymer that people have used since ancient times and is still widely active [1]. It is found in many creatures, from green leafy plants to primitive organisms such as seaweeds, flagellates, and bacteria [2]. Cellulose is also an important compound used in the production of artificial cellulose-based yarns, biofilms and various cellulose derivatives used for industry and in many similar industrial establishments. As it is a non-toxic, biodegradable polymer with high tensile and compressive strength, it is widely used in various fields such as pharmaceutical, food, cosmetics, textile, paint, and paper industry [3].

The coating is defined as a liquid or powder substance that provides color with a process that temporarily or permanently changes any crystal structure of the materials when applied on a surface [4]. Since coatings could be applied to almost all surfaces, it was used in many fields such as art, industrial coatings, design especially epoxy coatings [5]. Many raw materials, each with different properties, were used in the coating, which ensures the emergence of basic properties. The main components that made up the structure of the coating were binders, solvents, pigments, and additives [6]. The coatings could be categorized as water based and solvent based on solvent form. Water-based coatings that use water as solvent have fast drying time and very little odor and have a longer lifetime [7]. Coatings that are

thinned with solvents such as thinner during application are called solvent-based coatings. They need a long time to dry and have some harmful effects on the environment [8]. Global coating production was shifted from developed countries such as the EU and the USA to developing countries. Industrial coatings are consumed more than construction coatings in the world coating industry [9]. The progress of Turkey in harmony with technological developments has made it an important place in the world market within the scope of coating production activities. Production capacity in the coating sector in Turkey has been increasing from the past. As a result of this, Turkey is located on the sixth largest paint manufacturer in Europe [10].

Carrot is one of the important root vegetables, rich in bioactive compounds such as carotenoids and dietary fibers, containing many functional ingredients [11]. The sectors that produce carrot juice form a high amount of carrot pulp. Carrot pulp contains 17% of  $\alpha$ -carotenes and 31-35% of  $\beta$ -carotenes in fresh carrots [12]. The investigators were reported that the fiber components of carrot pulp (on a dry weight basis) were 3.88% pectin, 12.3% hemicellulose, 51.6% cellulose and 32.1% lignin. Therefore, the carrot pulp remaining after the water has been removed from the structure of carrots has been determined to be a compound with bioactive properties that can be discovered in the development of food ingredients and dietary

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supplements. When the carrot pulp was dried and examined, it was seen that it had 4-5% protein, 8-9% carbohydrate, 5-6% mineral and 37-48% fiber content. For this reason, carrot products are known to be a good source of fiber. Carrot pulp, which contains approximately 50% of carotenoids and important fibers, is a significant nutrient that can be used in the development of products with high nutritional value [11].

Volcanic lava stone is a stone obtained from a slag with a very porous structure, which is formed because of volcanic activities, the lava of the basic feature was exposed from the cavities due to the compression of volcanic rocks [13]. When chemical analysis of volcanic lava stone was made, it was determined that contains oxide compounds and was determined that it was a very rich structure in terms of silicon, iron, calcium, and potassium [14].

Perlite is a glassy volcanic rock belonging to the family of rhyolite surface rocks with a water content of 2-5% [15]. Expanded perlite is a white stone with a glassy structure formed by grinding the raw perlite extracted from the source and then heating up to 750-1200°C [16]. Perlite resources are common in the world, especially in countries where volcanic regions are intense. The raw perlite reserves in most countries are USA, Greece, Hungary, and Turkey is located. When the data on the amount of perlite production in the period from 2011 to the present are examined, it is concluded that this rate has increased worldwide. In 2018, a total of 4600 tons of raw perlite was produced worldwide. Perlite has a light, elastic, and fireproof structure. As it provides a good level of heat and sound insulation, it is used for binding purposes in building materials in the construction industry, in increasing the durability of cement, in plaster raw materials; Due to its porous structure, it is used in the agricultural sector to allow the plants to breathe in the soil and to enable the soil to absorb water better because it does not dissolve in any form. Perlite stone, which is very rich in silicon, also contains alkali elements such as aluminum, potassium, iron, calcium, magnesium. [17].

The main objective in optimization achieves the best result under the specified conditions by minimizing the required effort or maximizing the desired benefit [18]. Response Surface Method is a multivariate statistical optimization method was used in areas where various input variables affect the performance measures or quality characteristics of the product [19]. The purpose of the response surface method optimizes the response (output variable) affected by many independent variables (input

variable) [20]. The experimental design applied in various fields of science and industry plays an important role in the correct progress of the process and operation of the system. Therefore, to obtain a clear result, the experiments must be planned and designed sequentially, and the results obtained must be analyzed. The response surface method is one of the most preferred experimental designs in optimization processes. This method is very useful as it enables the evaluation of the effects of many factors' interactions with themselves and with each other on one or more response variables.

The response surface method is a widely used mathematical method to model and analyze a process in which the desired response is influenced by various variables, and it is mainly aimed at optimizing the response. In this method, the answers are called the dependent variable and the parameters affecting the experimental process are called the independent variable [21]. In the response surface method, the Box-Behnken design was preferred when the number of factors is low. The test combinations consist of the edges of the cubic model and the midpoints of the edges. In this design, the codes -1, 0 and 1 represent 3 levels of each factor [22].

In this study, cellulose-derived organic and other additives were added to the water-based organic coating and the effect of the additives in prepared coatings on viscosity was compared in detail. For this purpose, three different natural additives that carrot pulp, volcanic lava stone and perlite stone were added to the coating. Box Behnken Method was used as an optimization method to determine the binary relationship of the parameters. As a result of the physical tests were applied to the additive coatings, the effect on improving the physical properties of the coating of the additives and the responses of the coating under different conditions by thermal analyzes to coatings that containing additives were observed. The data obtained as a result of the tests were examined in comparison with the reference coating and were evaluated.

## Experimental

### Materials

Experiments were carried out using water-based coating (Silicone White Interior Wall Coating), carrot pulp, volcanic lava stone, perlite stone, iron plate (10 x 10 cm) and covering card. Viscometer (RM 100 Lamy Rheology), coating device (QUORUM), applicator, precision scale (AND GF-600) and magnetic stirrer with heater (MTOPS MS300 HS)

were used in the process of applying physical tests and analyzes to the reference and prepared coatings.

*Additive Preparation*

First, carrot pulp was obtained by removing water from carrot using a juice extractor. This pulp was sundried for a day. Most of the dried carrot pulp was powdered in the food processor. To prevent caking in the coating, it was passed through a 90-micron screen to separate it from some small particles. Volcanic lava stone and perlite stone were also cut into small pieces and passed through 90-micron screen and completely powdered (Fig. 1).



Fig 1: Additives in coating.

*Method*

In the experimental study, natural additives were added to the water-based coating in the determined proportions (0-6% by mass) carrot pulp, volcanic lava stone and perlite stone. Carrot pulp was added mainly to increase viscosity and it was aimed to improve the physical properties of the water-based coating along with other additives (Fig. 2). Design Expert package program was used to determine the proportions of additives added to the coating and achieve the best result with minimum number of experiments. To observe the effect of additives added to the coating on the physical properties of the coating in comparison with the reference coating, the Box-Behnken design method was used within the scope of the Design Expert Optimization Method. With this method, it was aimed to achieve optimum results by adding three additives (carrot pulp, volcanic lava stone, perlite stone) to the coating in three different percentages. In each experiment was carried out for this purpose, different additives were added to 5 grams of coating in the range of 0-6% by mass. A total of 17 experiments including 5 repetitions were performed by applying the 3-factor 3-level Box-Behnken design method. 3 levels in Box-Behnken design were coded as -1, 0, 1. Additional values were chosen equidistant

between these three levels. Codes were determined by the Box-Behnken design method were included in Table-1.

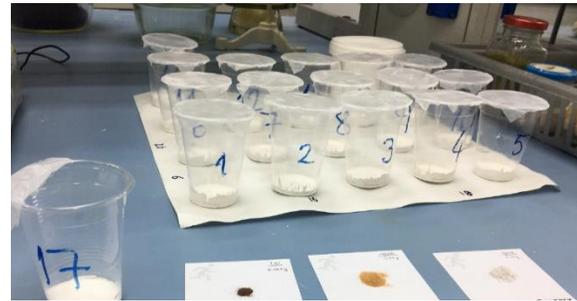


Fig. 2: Preparation of organic coatings with additives.

Table-1: 3-level (-1, 0, 1) 3-factor (A, B, C) Box-Behnken design and percentages of additives in organic coatings

No	Carrot pulp (A)	Volcanic lava stone (B)	Perlite stone (C)
1	0	1	-1
2	-1	-1	0
3	0	1	1
4	1	-1	0
5	0	0	0
6	-1	0	1
7	0	0	0
8	0	-1	-1
9	0	0	0
10	1	0	-1
11	0	0	0
12	0	-1	1
13	-1	0	-1
14	0	0	0
15	1	1	0
16	-1	1	0
17	1	0	1
Level	(%, w/w)	(%, w/w)	(%, w/w)
-1	0	0	0
0	1.5	1.5	1.5
1	3	3	3

*Characterization*

Physical and chemical tests (viscosity, hydrophobicity, corrosion, crosscut, heat transfer) were applied to determine how the additives added to the water-based coating affect the properties of the coating and compare the coating's properties with the reference coating. SEM (Zeiss EVO LS 10) and TG/DTA (SII 6000 Exstar TG/DTA 6300) analyzes were carried out to determine the characterization of additives added to the coating.

## Results and Discussion

### Design Expert Results

Box Behnken design was a very useful response surface method that provided a general information about the minimum number of experiments in a study, the experimental parameters and the errors that occurred in the experiment and their relationship with each other. As a result of the Box Behnken design, maximum information was obtained with a minimum number of experiments. In this method, there were 3 levels as (-1,0,1) corresponding to the factor number between 3-21. In the Box Behnken design, combinations where the factors were at the highest or lowest conditions at the same time were not observed. It was a very practical method to avoid experimental studies occurring under extreme conditions [23, 24]. As a result of applying the Box-Behnken design to the numerical data obtained from the experiments, different mathematical models such as linear, quadratic, cubic or 2FI (two-factor interaction) were formed. There were some terms used in this design. For example, A, B and C represented linear coefficients for independent variables, AB, AC and BC represented coefficient of binary interactive variables, A<sup>2</sup>, B<sup>2</sup> and C<sup>2</sup> represented quadratic coefficients. The correlation coefficient (R<sup>2</sup>) was used to express the relationship between variables and takes values between -1 and 1. As it approached 1, the accuracy of the experiment increased. The corrected R<sup>2</sup> was the correlation coefficient formed by removing the parameters that had no effect on the created model. Estimated R<sup>2</sup> was the correlation coefficient determined hypothetically by the model. The significance level of the differences between the averages and their relationship with each other was determined by performing an analysis of variance (ANOVA) [25].

Within the scope of experimental studies, statistical analyzes such as variance analysis, regression analysis and graphical representations of experimental responses were obtained using the Design Expert 7.0.0 package program. Parameters in the experiment set: carrot pulp (A), volcanic lava stone (B) and perlite stone (C). 3 levels for the 3 parameters in the experiment set was determined using the Box-Behnken design method. The levels were expressed in the design as -1, 0, 1 correspond to 0%, 1.5% and 3% of the parameters, respectively. The viscosity measurement results of the prepared additives were shown in Table-2.

Table-2: Viscosity measurement results.

No	Viscosity (mPa.s)	No	Viscosity (mPa.s)
1	1152	10	1859
2	872	11	1215
3	1462	12	1209
4	2013	13	1002
5	1103	14	1136
6	735	15	1834
7	1209	16	1096
8	1224	17	2159
9	1193	Reference	1008

The number 3 experiment was determined as the coating that was given the optimum result for the viscosity from the additive coatings. When the results were examined, it was seen that there was an increase in viscosity compared to reference coating. Variance analysis was carried out by Design Expert program to analyze the interaction of independent parameters with each other and effect on viscosity. When the results of variance analysis for viscosity of coating were examined in Table-3, the p value of model terms (AA, AB, AC, A<sup>2</sup>) containing carrot pulp (A) were less than 0.05 (p<0.05), p values for other model terms (BB, CC, BC, B<sup>2</sup>, C<sup>2</sup>) were appeared greater than 0.05 (p>0.05). These results were shown that carrot pulp was an important parameter affecting viscosity of coating both and in its interactions with other parameters, and volcanic lava stone (B) and perlite stone (C) was not significant effected on viscosity.

According to the experimental results, the regression model created for the viscosity of the coating was determined as quadratic. The R<sup>2</sup> value of the model was found to be 0.9844. This value indicates that the quadratic model could explain 98.44% of the change in viscosity. The estimated R<sup>2</sup> value was found to be 0.8041. This means that it could explain 80.41% of the changes that occur in the estimation of new observations. Corrected R<sup>2</sup> value was found to be 0.9644. This situation shows that 96.44% of the change in viscosity could be explained with this model. The difference between the corrected R<sup>2</sup> and the predicted R<sup>2</sup> was not much, and the coefficient of variation is low, indicating that this model is a good model for predicting new observations in the experiment. In model statistics, predicted and adjusted values were shown a good correlation (Fig 3).

The quadratic regression model created for viscosity was given as Eq.1:

$$R1 = +1171+(520xA)+(28xB)+(41xC)-(101xAB)+(142xAC)+(81xBC)+(230xAA)+(53xBB)+(38xCC) \quad (1)$$

The abbreviations (A, B, C, R1) in Equation 1 were represented as carrot pulp, volcanic lava stone, perlite stone and viscosity, respectively. In addition, AB, AC, BC, AA, BB and CC showed the binary interaction of parameters.

Table-3: Model Statistics Summary (Quadratic).

Source	Sum of Squares	df	Mean Square	F value	p-value
Model	2,581e+006	9	2,868e+005	49,12	<0,0001
A-A	2,162e+006	1	2,162e+006	370,3	<0,0001
B-B	6441,13	1	6441,13	1,1	0,3285
C-C	13448	1	13448	2,3	0,1729
AB	40401	1	40401	6,92	0,0339
AC	80372,25	1	80372,25	13,76	0,0076
BC	26406,25	1	26406,25	4,52	0,071
A <sup>2</sup>	2,221e+006	1	2,221e+005	38,03	0,0005
B <sup>2</sup>	11671,67	1	11671,67	2	0,2003
C <sup>2</sup>	6048,04	1	6048,04	1,04	0,3427
Residual	40872,55	7	5838,94		
Lack of Fit	31159,75	3	10386,58	4,28	0,0971
Pure Error	9712,8	4	2428,2		
Cor Total	2,622e+006	16			
Standard deviation	R <sup>2</sup>	Adjusted R <sup>2</sup>	Predicted R <sup>2</sup>	Coef. of Variation (%)	
76,41	0,9844	0,9644	0,8041	5,78	

Model	Std. Deviation	R <sup>2</sup>	Adjusted R <sup>2</sup>	Predicted R <sup>2</sup>	Variation Coefficient (%)
Quadratic	76,41	0,9844	0,9644	0,8041	5,78

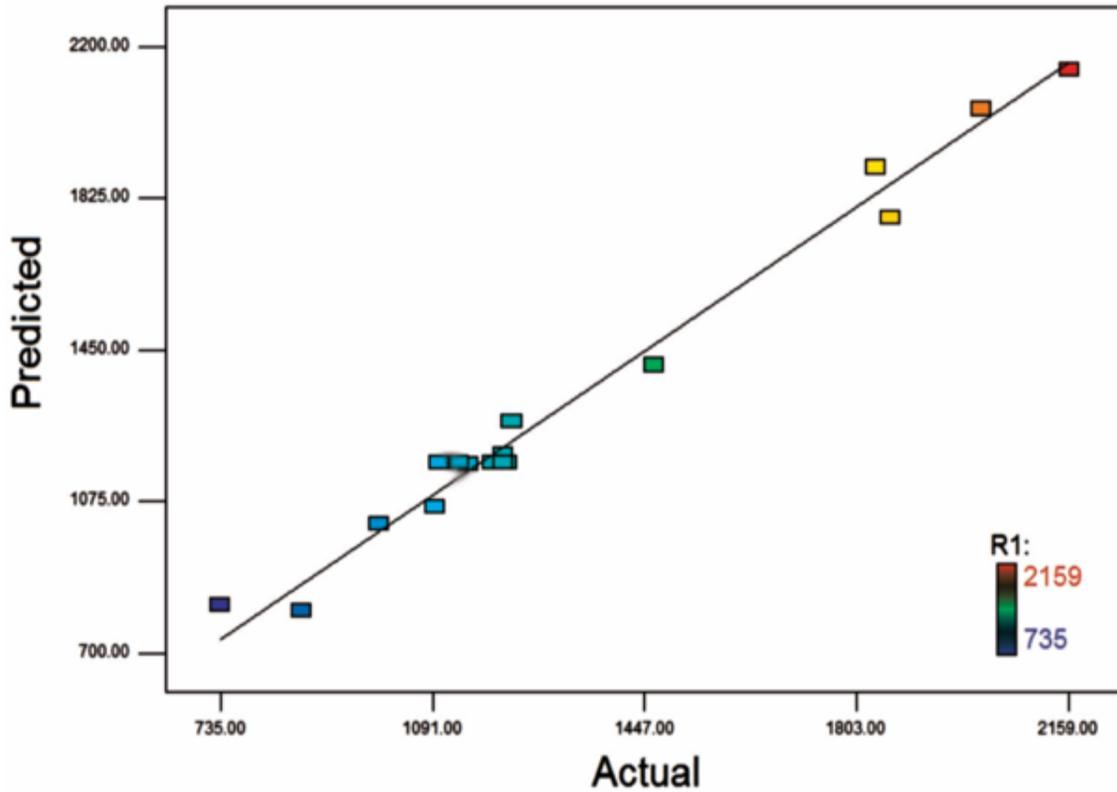


Fig. 3: Comparison of predicted and actual values.

3D graphics showing the effect of additives added to the coating on the viscosity of the coating

were given in Fig 4. When the graphics were examined, it was seen that there was a direct

proportion between the amount of carrot pulp in the coating and the viscosity value. When the numerical data were compared, it was confirmed that the optimum coating with carrot pulp has a higher viscosity than the reference coating. From here, it could be interpreted that carrot pulp was an important parameter with cellulose content that increases the viscosity of the coating, while volcanic lava stone and perlite stone were not very effective in increasing viscosity.

*Test Results*

*Crosscut, corrosion and hydrophobicity tests*

In cross-cut test, an adhesive tape was applied to the cross pattern formed on the coatings for iron and aluminum surfaces. It was seen that the prepared water-based organic coatings with additives could be applied to metal surfaces such as iron and aluminum in many different application areas, especially in the construction sector. The adhesion test results showed that when the prepared additive organic coatings were compared with the reference organic coatings, it was seen that the adhesion strength of the water-based organic coatings with additives applied to the surface was quite strong and did not leave the surface, thus

protecting the surface in different conditions (Fig 5). For corrosion test, optimum and reference coatings were kept in 5% sodium chloride solution for 2 hours. Afterwards, they were placed in the water vapor cabin and left in a closed environment for 24 hours. At the end of the period, the corrosive spots detected on the surface of the coating applied to the iron plates were evaluated visually. When the results were examined, it was found that the optimum coating protected the metal surface against to corrosion in case of comparing with reference organic coatings. When the visual results were examined, it was determined that corrosion points were formed in approximately one quarter of the iron plate on which organic coating was applied, while this rate was higher in the reference organic coating. This result was an indication that the 3% by weight volcanic lava stone added to increase the corrosion resistance of the coating was effective in reducing the corrosion. Because of hydrophobicity test, it was observed that both the optimum and the reference coating was not absorbed the water, but that they were water repellent. This situation was an indication that the additives were added to the coating was not adversely affected the hydrophobicity of the coating. Although different additives were added to the coating, the coating was remained hydrophobic (Fig 5).

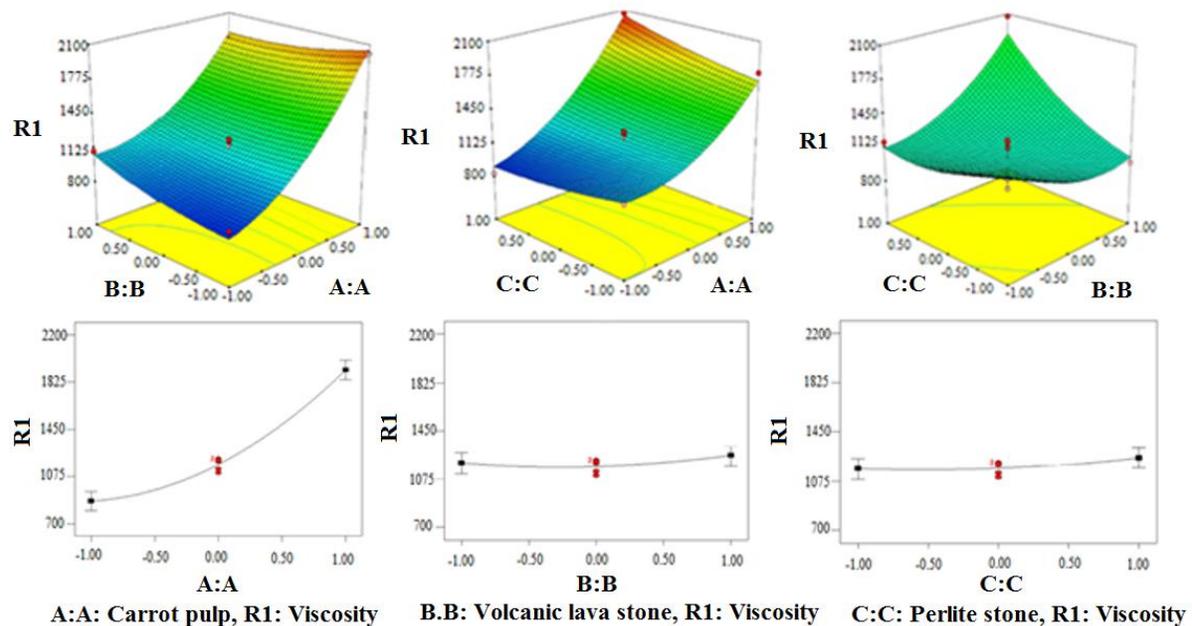


Fig. 4: 3D graphs with change of viscosity (A:A: Carrot pulp, B:B: Volcanic lava stone C:C: Perlite stone, R1: Viscosity).

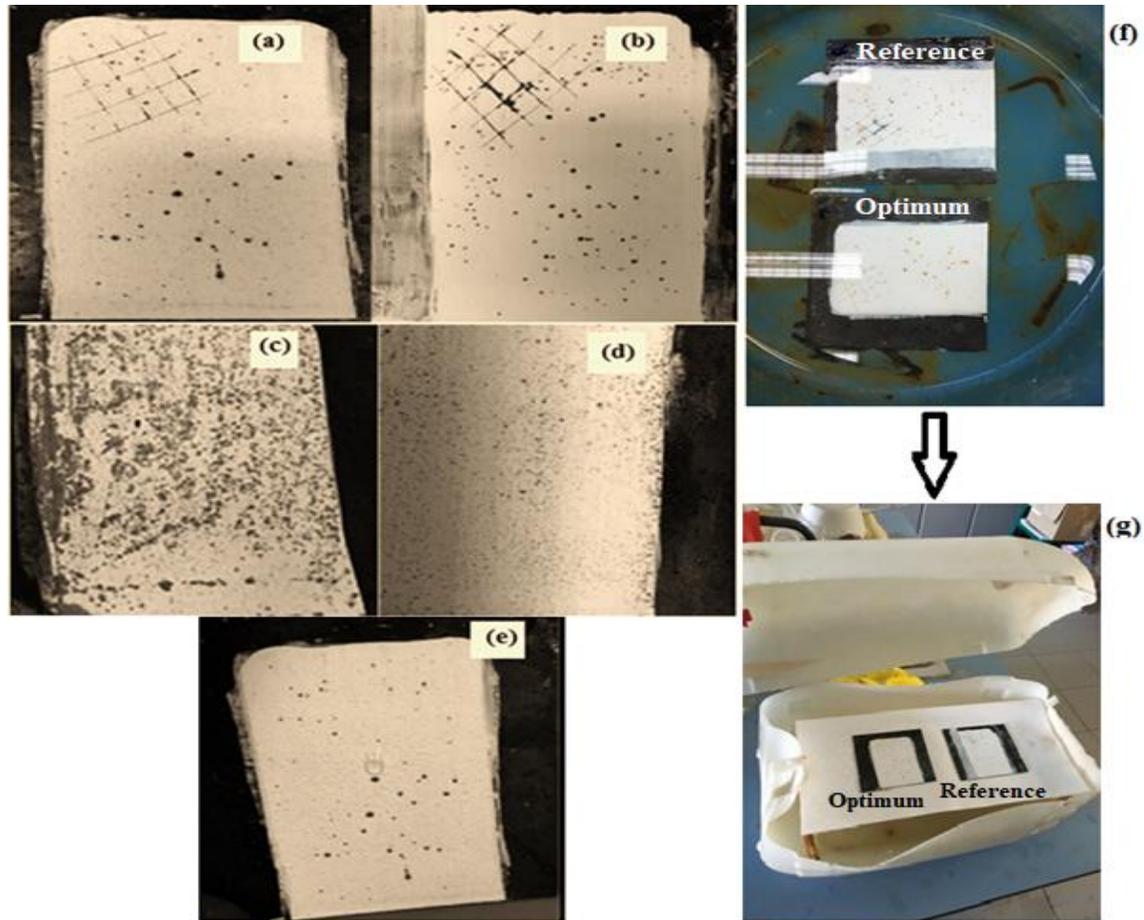


Fig. 5: Test results: a) Cross-cut test of optimum coating, b) Cross-cut test of reference coating, c) Corrosion test of reference coating, d) Corrosion test of optimum coating, e) Hydrophobicity test of optimum coating, f) Metal plates before corrosion of optimum and reference coatings, g) Metal plates after corrosion of optimum and reference coatings.

#### TG/DTA Results

TG/DTA analysis was carried out to examine the changes in mass losses due to the increase in temperature with nitrogen gas application on the optimum and reference coating samples (Fig 6).

When the analysis result of the optimum coating containing carrot pulp, volcanic lava stone and perlite stone additives was examined, it was seen that there were three curves, TG, DTG and DTA. In the TG curve, the mass loss occurring in the coating sample at 25-800 °C was shown. Until the temperature was about 220 °C, a 3.4% reduction in mass occurred. This was due to the removal of volatile substances in the coating structure. The highest mass loss occurred between 220-560 °C with a rate of 34.8%. It appears that an endothermic curve was formed in this range. The degradation temperature of the optimum coating structure from the DTA curve was 409,3°C. When the analysis result of the reference coating was examined,

it was seen that there was a mass loss of 2.6% between 25-300°C and 5.4% between 420-480 °C. Low mass losses occur because of the removal of volatile components in the coating structure. The highest mass loss was between 300-420°C with 26.6%. At the same time, the curve formed between 300-420 °C was endothermic. The actual decomposition temperature of the reference dye from the DTA curve was read from the graph as 410°C. When comparing the reference coating with the optimum coating, it was seen that the decomposition temperatures were very close to each other. This was an indication that carrot pulp, volcanic lava stone and perlite stone additives added to the optimum coating was not caused any decrease in the decomposition temperature of the coating and was not caused degradation in coating structure. As a result, although more than one additive was added to the coating, the structure of the coating was preserved.

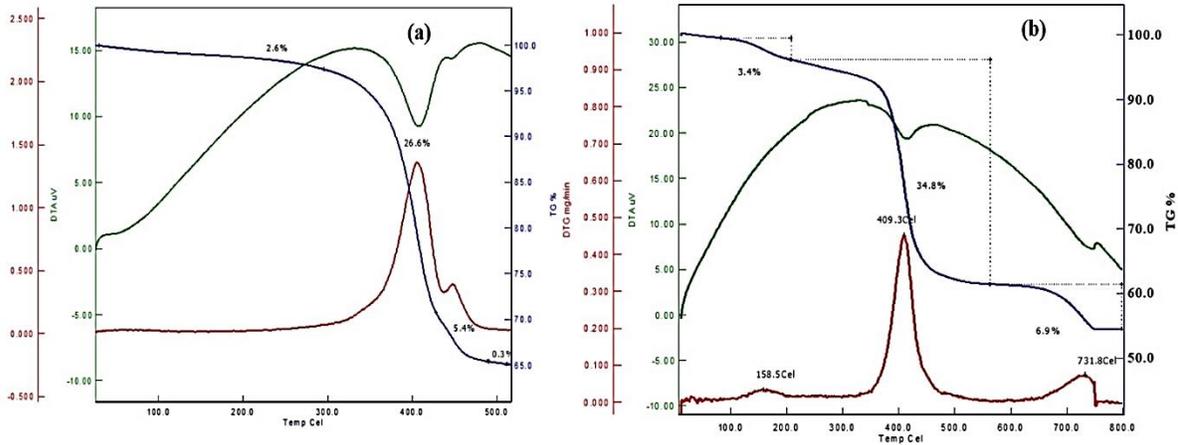


Fig. 6: TG/DTA analysis results a) Reference coating, b) Optimum coating.

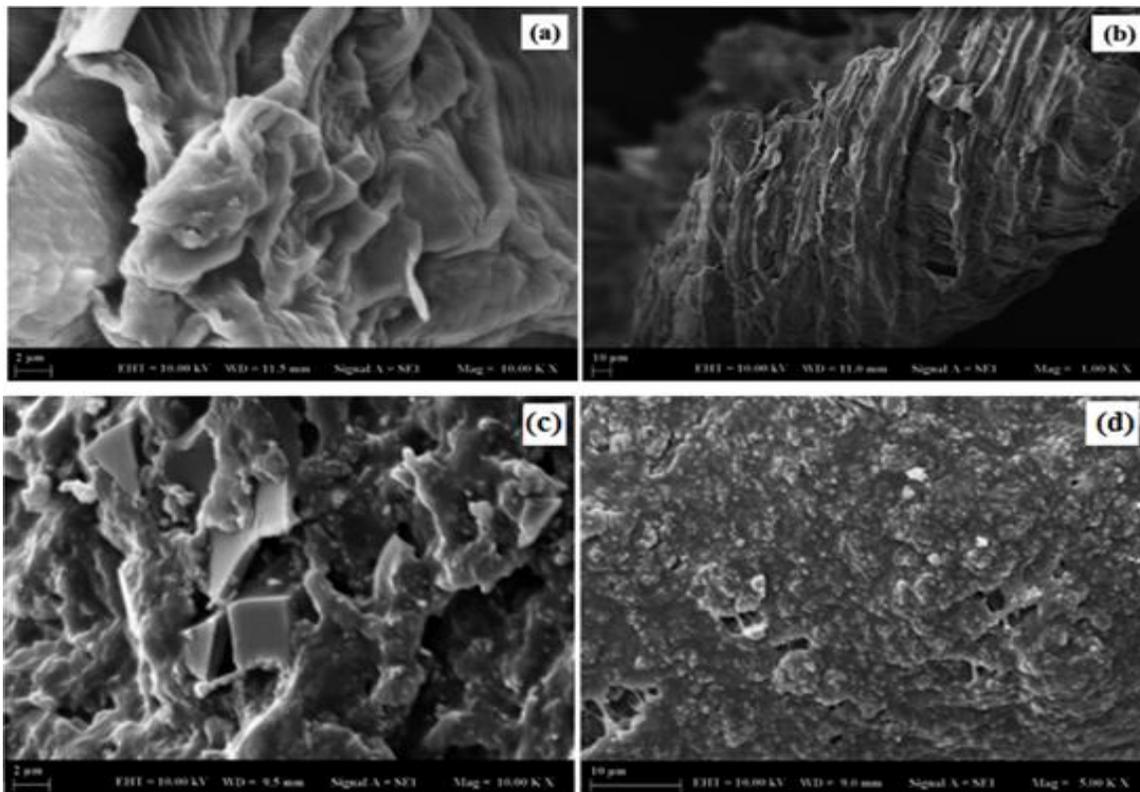


Fig. 7: SEM images: Carrot pulp a) 10000X magnification, b) 1000X magnification; Optimum coating c) 10000X magnification, d) 5000X magnification.

#### SEM Results

Internal structure of carrot pulp and doped optimum dye molecules were investigated by using Scanning Electron Microscope in SEM analysis. First of all, the samples were gold plated. Thanks to the gold coating applied, further enlargements were made and

micro-sized structures of the samples were obtained. SEM images of optimum coatings were given in Fig 7.

When looking at the images obtained from the analysis of powdered carrot pulp, it was seen that there were more than one long strands of fibers in the structure of the carrot. There were some small size particles in different sized images. It was seen that the additives in the coating molecule applied at a

magnification of 5000 times show a homogeneous distribution in the coating and it has a smooth structure. It was determined that there were small particles in the coating at 10000 times magnification. In this magnification, where smaller images were obtained, it was observed that the additives (carrot pulp, volcanic lava stone, perlite stone) made the structure of the coating tighter and more intense.

### Heat Transfer Results

In experimental studies, perlite stone was added to the coating to improve the emissivity of the coating by making use of the heat transfer by radiation. Within the scope of heat transfer test, an experimental setup was established to determine emissivity values by using the heat transfer with the reference and optimum coatings prepared and to compare them. The coatings were placed on the heated magnetic stirrer, respectively, and the surface temperatures were measured for equal periods with the sensor.

For radiant heat transfer, the emissivity of the aluminum plate and then the reference and optimum coating were calculated. The results obtained were given in Table 4.

Table-4: Results obtained in radiant heat transfer experiment ( $\epsilon$ : Measured emissivity)

$\dot{Q}_{radiation} = \epsilon \cdot \sigma \cdot A \cdot (T_s^4 - T_c^4)$			
Material	Temperature (K)	Area (m <sup>2</sup> )	$\epsilon$
Aluminumplate	348,15	0,03250	0,50
Reference coating	317,15	0,00636	0,81
Optimum coating	308,15	0,00636	0,71

When the results were evaluated, the emissivity of the optimum coating was lower than the reference coating. In literature, the emissivity value of white coating was given as 0.93 [26]. The different result was due to the inability to prevent heat losses, however insulated during the experiment. This situation was interpreted as being effective in reducing the heat transfer of perlite stone added to the coating for the purpose of insulation.

### Conclusion

As a result, it was found that carrot pulp was a high cellulose content in water-based coatings, thus it increased the viscosity significantly when added to the coating. While the viscosity of the reference coating was 1008 mPa.s, the viscosity of the coating obtained as 1462 mPa.s by means of the organic additives added to the coating. Because of the visual examination of the hydrophobicity property of the organic coating, it was concluded that the water

droplets flowed without wetting the surface and the contact angle between the surface and the droplet was over 90 degrees considering the contact angle, which was the criterion of hydrophobicity. The measured emissivity value of the organic coating was found to be 0.71. This result revealed that the organic coating prepared with the addition of additives has a better ability to absorb heat on the surface compared to the reference coating. The experiment number 3 with three additives in the organic coating (1.5%-carrot pulp, 3%-volcanic lava stone, 3%-perlite stone) was determined as the optimum coating and the test results applied for this experiment revealed that the properties of organic coating could be improved and evaluated for different industrial applications.

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