กรมชลประทาน

บทความ ทางวิชาการ

Formulation of Tile Adhesive Using Statistical Mixture Design

สำนักวิจัยและพัฒนา

นายจีรภัทร์ เตชะกุลชัยนันต์
กลุ่มวิจัยและพัฒนาด้านวิศวกรรม
สำนักวิจัยและพัฒนา กรมชลประทาน

ตุลาคม 2552

FORMULATION OF TILE ADHESIVE USING STATISTICAL MIXTURE DESIGN

Jeerapat Techakunchaiyanunt[1] and Thanakorn Pheeraphan[2]

[1]*Graduate student, Asian Institute of Technology, Pathumthani 12120, Thailand and Civil Engineer, Research and Development Department, Concrete and Material Section, RID, Tiwanon Road, Nonthaburi 11120, Thailand, e-mail: T.jeerapat@hotmail.com*

[2]*Department of Civil Engineering, Royal Thai Air Force Academy, Paholyothin Road, Saimai, Bangkok 10220, Thailand and School of Engineering and Technology, Asian Institute of Technology, Pathum Thani 12120, Thailand, e-mail:petevmi@alum.mit.edu*

ABSTRACT : In this paper the statistical mixture design with upper and lower bounds of component proportions was adopted to study the tensile adhesion strength of tile adhesive made with ordinary Portland cement, redispersible polymer powder, cellulose ether, and sand. Based on the results of a minimum of seventeen design points, four second-degree mixture models or quadratic models were used to establish the tensile adhesion strength predicting equations at different curing conditions according to EN-1346 and EN-1348. For each formulation, the prediction using a formula derived from the relationship between the predicted value and experimental value was within 36.5% of experimentally measured values. This was supported by the comparison of the calculated and tested tensile adhesion strengths.

KEYWORDS : Dry mortar, Tile adhesive, Statistical Mixture Design, Prediction Equation Model.

1. Introduction

In the simplest case, thick bed mortars are a mixture of water, Portland cement, and fine aggregate. Additional components, such as organic binder (redispersible polymer powder) and additive (cellulose ether), may be added to the basic mixture to enhance certain properties of the fresh or hardened tile adhesive. High performance tile adhesive, which may be required to meet several performance criteria (e.g., tensile adhesion strength, slip resistance) simultaneously, typically contain at least four components [1].

In order to develop tile adhesive formulations, the trial and error or one variable at a time method requires many experiments and there is no guarantee that an optimal formulation can be achieved. Moreover, the interaction between different factors, which can influence the several target responses, may not be detected. Statistical mixture design is useful tool and give good prediction equation model of response in the many fields such as chemical industry, food science, glasses and ceramics, pharmacy [2-8]. It has proven, in all cases studied, to lead to greater efficiency in the results obtained, and to be less demanding in time and material. Furthermore, statistical mixture design has not been applied in the field of tile adhesive dry mortar, and also development of tile adhesive dry mortar by using statistical mixture design will gain knowledge to improve high performance tile adhesive.

This study work is intended to establish prediction equation model relating the responses to develop high performance tile adhesive with the proportions of four raw materials which are ordinary Portland cement, redispersible polymer powder, cellulose ether, and sand using the statistical mixture design.

2. Statistical Mixture Design

2.1 Mixture Design

A mixture problem is mainly concerned with mixture development research, such as formulation modeling and optimization. The tile adhesives raw materials mixture components or variables in a mixture system are dependent on each other. This leads to the fundamental difference of the statistical experimental methodology applied in a tile adhesives mixture system from that of a tile adhesives process system. In a mixture system a specific relationship exists between all n ingredients such as shown in Equations (1) and (2). This means that no component variable can be changed without changing simultaneously any of the other component variables.

$$\sum_{j=1}^{n} x_j = 1 = 100\% \qquad (1)$$

$$\text{or } X_j = 1 - \sum_{i=1}^{j-1} x_j - \sum_{i=j+1}^{n} x_i \qquad (2)$$

2.2 Model Forms

The problem of mixture development actually focuses on the modeling of a mixture system based on some limited experiments. Let us assume that there is a mixture in which a functional relationship exists between the n component $X_1, X_2, X_3, ..., X_4$ and a tile adhesives quality index Y would be related in continuous fashion to mixtures comprised of $X_1, X_2, X_3, ..., X_4$ and considered to be response surface. Equation (3) exactly describes the response surface:

$$Y = f(X_1, X_2, X_3, ..., X_4) \quad (3)$$

2.3 Extreme Vertices Design

The composition of most multicomponent mixture systems is restricted by upper or lower or both boundary condition. A researcher may in practice often be faced with multicomponent mixtures where definite limitations are imposed on ratios of individual components as shown in Equation (4):

$$0 \le a_i \le X_i \le b_i \le 1 \quad (4)$$

$$\sum_{i=1}^{q} a_i \ge 1; \sum_{i=1}^{q} b_i \le 1 \quad (5)$$

where a_i and b_i correspond to upper and lower limit ratios of the i-th component.

To reduce the scope of calculations and to formalize the approach to the choice of design points of a design of experiment, McLean and Anderson [9] suggested this procedure:

2.3.1 All the possible combinations of the two levels a_i and b_i, are put down for each and every component, but in each combination the content of one component is omitted. The number of these combinations for a q-component mixture is $q.2^{q-1}$.

2.3.2 Among all the combinations those are selected whose sum of components is less than one and that meet the limitations of Equation (5). Into the combinations selected the omitted components are added in amounts defined by the relationship $\sum_{i=1}^{q} X_i = 1$. The design points thus obtained and satisfying Equation (5) lie at vertices of the bounding polyhedron.

2.3.3 To the design points obtained are added center points (centroids) of two-, three-,..., and $(q-1)$ -dimensional faces of the polyhedron and its center point. Coordinates of a central point are determined by taking average coordinates of previously chosen vertices.

For a four components system (n=4), the design points can be found as shown in Figure1.The relation between the performance index or responses Y and compositions X_i of n component system can be regressed by a second-degree mixture model or quadratic model in Equation (6).

$$Y = \sum_{j=1}^{n} B_j.X_j + \sum_{j<k=2}^{n} B_{jk}.X_j.X_k \quad (6)$$

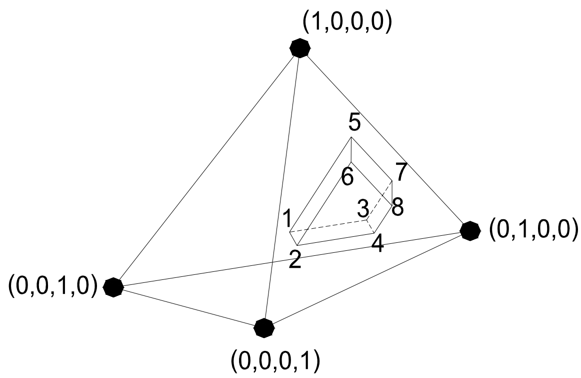

Figure 1 For q=4: The restricted experimental region for the flare experiment, with its extreme vertices numbered 1-8, giving easy identity to the six faces.

3. Experimental Program

3.1 Materials

The following raw materials were used: Ordinary Portland Cement (Type I), redispersible polymer powder were provided that had vinyl-acetate/ethylene copolymer as a main composition of redispersible polymer powder, cellulose ether that had Methyl hydroxyethyl cellulose modified as a main composition of cellulose ether, Natural river sand retaining sieve No.100 (300 µm) was used as fine aggregate and all sand were cleaned and dried. The unglazed porcelain tile sizing 30 mm x 30 mm was cut to be the facial dimensions of 50 ± 1 mm x 50 ± 1 mm. Its water absorption is 0.20%. The concrete substrate sizing 100 mm x 300 mm x 25 mm was used.

3.2 Preparation of tile adhesives

Four constituents, Ordinary Portland Cement (x_1), Redispersible Polymer Powder (x_2), Cellulose Ether(x_3), Natural river sand (x_4), were mixed according to mixture design, with multiple constraints on the component proportions, using fixed intervals, as in Table 1.The total amount of each batch was 2000 g.

Commercially available computer software for experiment design was used to design and analyze the experiment. The program selected 17 points from a list of candidate points that is known to include the best points for fitting the special quadratic equations. Mixture design-extreme vertices design was chosen to ensure the design selected could estimate the quadratic model while spreading points as far away as possible from one another. Furthermore, the selected composition system in this investigation was ordinary Portland cement, sand, redispersible polymer powder, cellulose ether with intervals mentioned above. The 17 experimental points were generated and randomly arranged by Design Expert software program (Version 7.1.6 trial) [10] and the respective tile adhesive formulations are described in Table 2**.**

Table 1 Variables and intervals selected to perform the mixture design

Composition	ID	Fraction range (% by weight)	
		Minimum	Maximum
Fraction of ordinary Portland cement	x_1	20.00	50.00
Fraction of sand	x_2	50.00	80.00
Fraction of Redispersible polymer powder	x_3	1.00	10.00
Fraction of cellulose ether	x_4	0.10	0.50

x_1+x_2+x_3+x_4=100%of the mixture design ($\sum_{i=1}^{n} X_i = 1$)

Table 2 The percentage and composition of each component in each formulations

Batch No.	Run order	Percentage of compositions (% wt)			
		C	S	RP	CE
1	10	0.2000	0.7890	0.0100	0.0010
2	11	0.2000	0.7850	0.0100	0.0050
3	16	0.2000	0.6990	0.1000	0.0010
4	9	0.2000	0.6950	0.1000	0.0050
5	17	0.4890	0.5000	0.0100	0.0010
6	8	0.4850	0.5000	0.0100	0.0050
7	2	0.3990	0.5000	0.1000	0.0010
8	4	0.3950	0.5000	0.1000	0.0050
9	13	0.3220	0.6220	0.0550	0.0010
10	6	0.3200	0.6200	0.0550	0.0050
11	5	0.3435	0.6435	0.0100	0.0030
12	14	0.2985	0.5985	0.1000	0.0030
13	7	0.2000	0.7420	0.0550	0.0030
14	12	0.4420	0.5000	0.0550	0.0030
15	3	0.3210	0.6210	0.0550	0.0030
16	15	0.3210	0.6210	0.0550	0.0030
17	1	0.3210	0.6210	0.0550	0.0030

3.3 Mixing and Casting & Curing

3.3.1 Mixing and Casting

A quantity of 2 kg of the tile adhesive was prepared in a mixer. The following procedure was found to be more effective. Firstly, tile adhesive were dry-mixed for 30 seconds with the mixer operating at low speed setting and half of the mixing water was added during the next 30 seconds of mixing. Then, the remainder of the mixing water was added and mixing was continued for a further 60 seconds. After that, the mixer was stopped and the paste was scraped from the sides of bowl before mixing at high speed setting for 60 seconds. Secondly**,** apply a thin layer of tile adhesive to the concrete slab with a straight edge trowel by using the notched trowel having 6 mm x 6 mm was held at an angle of approximately 60 degree to the substrate a right angle to one edge of the slab. Then, porcelain tiles are placed after 5 minutes onto the adhesive mortar at a distance apart of 50 mm and load each tile with 2-kg weight for 30 seconds.

3.3.2 Curing

1)28-day tensile adhesion strength after dry curing

After 27 days storage under standard conditions, bond the pull- head plates to the tiles with epoxy. After a further 24-hour storage under standard conditions, determine the tensile adhesion strength of the tile adhesive by applying a force at a constant rate.

2)28-day tensile adhesion strength after wet curing

Condition the test units in standard conditions for 7 days and immerse in water at the standard temperature. After 20 days remove the test specimen from the water, wipe with cloth and bond the pull-head plates to the tiles. After a further 7 hours, immerse the test specimen in water at standard temperature. On the following day remove the test specimens from water and immediately carry out the tensile adhesion test of the tile adhesive by applying a force at a constant rate.

3)28-day tensile adhesion strength after heat curing

Condition the test units in standard conditions for 14 days and then place the specimens in an air circulation oven at $70 \pm 2^{o}C$ for further 14 days. Remove from the oven and bond the pull-head plates to the tiles with epoxy. Condition the test specimens for a further 24 hours in standard conditions. Determine the tensile adhesion strength of the tile adhesive by applying a force at a constant rate.

4)28-day tensile adhesion strength after open time 20 minutes

After 20 minutes place on the adhesive and load each tile with 2 kg for 30 seconds. After 27-days storage under standard conditions, bond the pull- head plates to the tiles with epoxy. After a further 24-hours storage under standard conditions, determine the tensile adhesion strength of the tile adhesive by applying a force at a constant rate.

3.4 Evaluation of Formulation Properties

3.4.1 Tensile adhesives strength

Tensile adhesion strength test was measured in the different storage which are dry curing condition, wet curing condition, heat curing condition with EN1348[13], and open time 20 min according with EN1346[12] and EN12004 European standards[14]. In addition, the test will be conducted to determine the tensile adhesion strength of each mix proportions at the age of 28 days. The measurement for tensile adhesion strength was taken for ten samples and the mean was reported as a result. The tensile adhesion strength for each set of conditions will be determined as follow, first of all, determine the mean of the ten values. Secondly, discard the values falling outside the range of $\pm 20.00\%$ from the mean value and if five or more than five values remain, determine the new mean value, but if less than five values remain, repeat the test. To calculate the tensile adhesion strength were calculated from Equation (7):

$$f_t = \frac{L}{A} \qquad (7)$$

where:

f_t = The individual tensile adhesion strength in Newtons per square millimeters[N/mm^2 or MPa];

L = Total load in Newtons [N];

A = The bonding area in square millimeters[mm^2].

3.4.2 Water content

The water content of the paste was measured using a flow table tests. In addition, the flow table tests were used to control workability as indicated by flow value of mortar with the procedure given in Practice ASTM C305. The flow table value of all paste mixes was maintained at 180 ± 10 mm. It was calculated % Flow Table Value from Equation (8) and % Water demand from Equation (9):

$$\text{\% Flow Table Value} = \left(\frac{A1 - A0}{A0}\right) x100 \qquad (8)$$

where:

A_1 = Average of four readings in millimeters[mm^2];

A_0 = Original inside base diameter in millimeters[mm^2].

$$\text{\% Water demand} = \left(\frac{W_{water}}{W_{total}}\right) x100 \qquad (9)$$

where:

W_{water} = Weight of water measuring in grams[g];

W_{total} = Weight of total dry mortar in grams[g].

3.5 Data analysis

The data of tile adhesive properties were used to evaluate for the response models as a prediction equations. Furthermore, all statistical parameter values were calculated by using Design Expert (Versin7.1.6 Trial) software program[10]. According to statistical analysis, there are two main steps which are fitting models step and model adequacy checking step. To consider fitting models step, assumption to select appropriate model is if test for significance of regression model using analysis of variance is significant, there are regression model that can represent the data and then not only the adjusted coefficient (Adj-R^2) were calculated but also comparison of the adjusted coefficient (Adj-R^2). After selected the appropriate models, model adequacy checking step was used to validate the model by checking lack of fit test. According to lack of fit assumption, if regression model was checked by using analysis of variance (lack of fit) is non significant, those regression model can be used to predict the data.

4. Experimental Results

The average value for 28-day tensile adhesion strength after dry curing, 28-day tensile adhesion strength after wet curing, 28-day tensile adhesion strength after heat curing, 28-day tensile adhesion strength after open time 20 min passed for each batch are shown in Table 3. The statistical analysis is described in detail for 28-day tensile adhesion strength after dry curing and the analyses for the other properties were performed in a similar manner.

4.1 28-day tensile adhesion strength after dry curing.

The 28-day tensile adhesion strength predicting equation models based on the 1-17 test batches is shown in Equation (10):

$$Y_1 = -1.21E{+}00x_1 - 2.09E{-}01x_2 + 6.52E{+}01x_3 - 2.25E{+}04x_4 + 5.31E{+}00\, x_1x_2 - 4.76E{+}01x_1x_3 + 2.27E{+}04\, x_1x_4 - 6.77E{+}01x_2x_3 + 2.26E{+}04x_2x_4 + 2.10E{+}04x_3x_4 \qquad (10)$$

where Y_1 is the 28-day tensile adhesion strength after dry curing and x_1, x_2, x_3, and x_4 are weight proportions of ordinary Portland cement, sand, redispersible polymer powder, and cellulose ether, respectively.

The predicted values of tensile adhesive strength after dry curing through the mixture design are compared with correspondent experimental values, as are showing predicting accuracy shown in Figure 2. For the range of experimental values for 28-day tensile adhesion strength after dry curing between 0.460 and 1.760 MPa, it was found that the maximum percentage of error was within ± 16%.

Table 3 Experimental values of tile adhesive properties of seventeen experimental points

Batch No.	Tensile adhesion strength (MPa)				Water Demand (%)
	Dry	Wet	Heat	Open time 20 min	
1	0.56	0.57	0.02	0.24	0.21
2	0.46	0.38	0.05	0.55	0.34
3	1.14	1.00	0.58	0.25	0.23
4	0.65	0.38	0.54	0.76	0.37
5	0.76	0.79	0.30	0.30	0.20
6	0.94	0.72	0.59	1.07	0.33
7	1.76	1.20	0.93	0.20	0.22
8	1.19	0.89	0.74	1.05	0.35
9	1.05	0.80	0.69	0.41	0.21
10	0.82	0.64	0.42	0.83	0.34
11	1.06	0.90	0.18	1.10	0.27
12	1.23	0.63	1.03	0.64	0.29
13	0.71	0.54	0.28	0.97	0.29
14	1.27	0.84	0.89	1.07	0.28
15	0.87	0.56	0.40	0.91	0.28
16	1.02	0.72	0.41	0.85	0.29
17	0.87	0.67	0.49	1.31	0.28

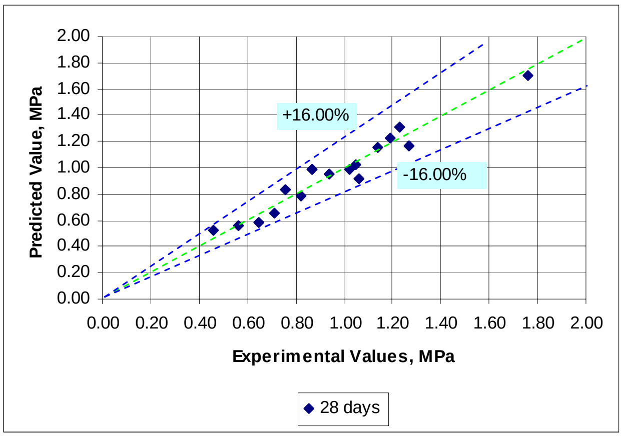

Figure 2 Relationship between predicted values and experimental value of tensile adhesion strength after dry curing.

4.2 28-day tensile adhesion strength after wet curing.

The 28-day tensile adhesion strength predicting equation models based on the 1-17 test batches is shown in Equation (11):

$$Y_2 = 1.64E+00x_1+3.98E-01x_2+2.66E+00x_3-6.65E+01x_4 \quad (11)$$

where Y_2 is the 28-day tensile adhesion strength after wet curing and x_1, x_2, x_3, and x_4 are weight proportions of ordinary Portland cement, sand, redispersible polymer powder, and cellulose ether, respectively.

The predicted values of tensile adhesive strength after wet curing through the mixture design are compared with correspondent experimental values, as are showing predicting accuracy shown in Figure 3. For the range of experimental values for 28-day tensile adhesion strength after wet curing between 0.380 and 1.200 MPa, it was found that the maximum percentage of error was within ± 28.5%.

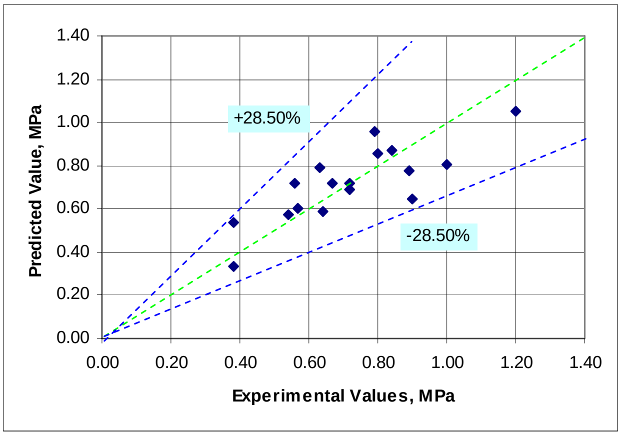

Figure 3 Relationship between predicted values and experimental value of tensile adhesion strength after wet curing.

4.3 28-day tensile adhesion strength after heat curing.

The 28-day tensile adhesion strength predicting equation models based on the 1-17 test batches is shown in Equation (12):

$$Y_3 = 1.02E+00x_1+0.39843x_2+6.69E+00x_3+6.81E+00x_4 \quad (12)$$

where Y_3 is the 28-day tensile adhesion strength after heat curing and x_1, x_2, x_3, and x_4 are weight proportions of ordinary Portland cement, sand, redispersible polymer powder, and cellulose ether, respectively.

The predicted values of tensile adhesive strength after heat curing through the mixture design are compared with correspondent experimental values, as are showing predicting accuracy shown in Figure 4. For the range of experimental values for 28-day tensile adhesion strength after heat curing between 0.180 and 1.030 MPa, it was found that the maximum percentage of error was within ± 33.5%.

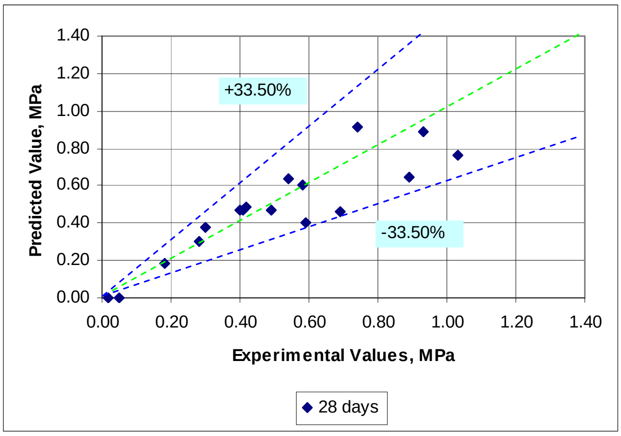

Figure 4 Relationship between predicted values and experimental value of tensile adhesion strength after heat curing

4.4 28-day tensile adhesion strength after open time 20 minutes.

The 28-day tensile adhesion strength predicting equation models based on the 1-17 test batches is shown in Equation (13):

$$Y_4 = -9.62E\text{-}01x_1 - 3.25E\text{-}01x_2 - 4.72E{+}0x_3 - 8.68E{+}04x_4 + 1.36E{+}00x_1x_2 + 4.73E{+}01x_1x_3 + 8.78E{+}04x_1x_4 + 5.24E{+}01x_2x_3 + 8.73E{+}04x_2x_4 + 8.80E{+}0x_3x_4 \quad (13)$$

where Y_4 is the 28-day tensile adhesion strength after open time 20 minutes and x_1, x_2, x_3, and x_4 are weight proportions of ordinary Portland cement, sand, redispersible polymer powder, and cellulose ether, respectively.

The predicted values of tensile adhesive strength after open time 20 minutes through the mixture design are compared with correspondent experimental values, as are showing predicting accuracy shown in Figure 5. For the range of experimental values for 28-day tensile adhesion strength after open time 20 min between 0.200 and 1.310 MPa, it was found that the maximum percentage of error was within ± 36.5%.

4.5 % Water Demand.

The %Water Demand predicting equation models based on the 1-17 test batches is shown in Equation (14):

$$Y_5 = 1.45E\text{-}01x_1 - 1.83E01x_2 + 4.05E01x_3 + 3.28E{+}01x_4 \quad (14)$$

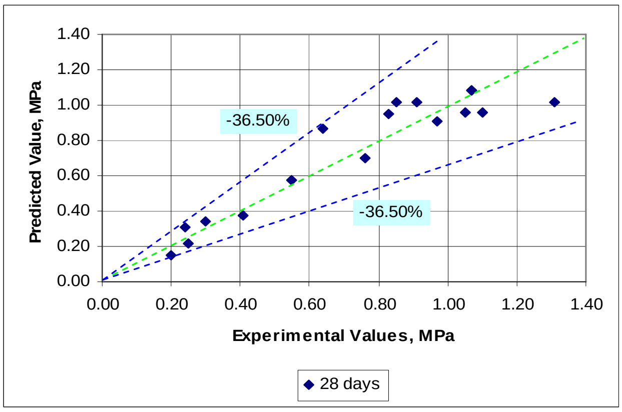

Figure 5 Relationship between predicted values and experimental value of tensile adhesion strength after open time 20 minutes.

where Y_5 is the %Water Demand of tile adhesives and x_1, x_2, x_3, and x_4 are weight proportions of ordinary Portland cement, sand, redispersible polymer powder, and cellulose ether, respectively.

The predicted values of water demand are compared with correspondent experimental values as predicting accuracy can be shown in Figure 6. For the range of experimental values for %water demand between 0.201 and 0.350%, it was found that the maximum percentage of error was within ± 3.00%

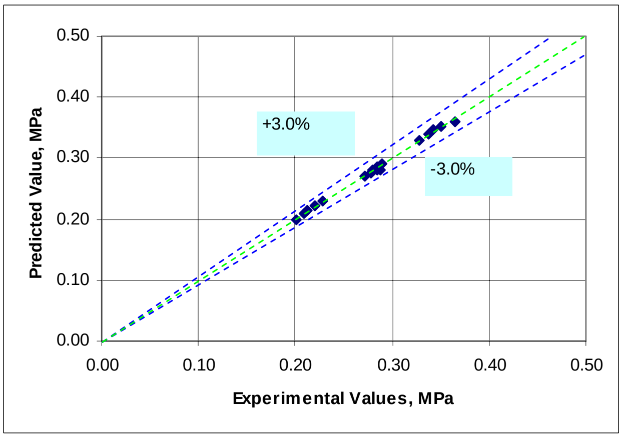

Figure 6 Relationship between predicted values and experimental value of percentage of water demand of tile adhesive.

5. Discussion

The accuracy of this prediction method significantly relies on two main factors which are workmanship, and curing condition. In order to test tensile adhesion strength of tile adhesive, there are many step of testing and it is quit complicate to control many step of testing to be uniform. Therefore, it is necessary to control the process during establish a formula to predict tensile adhesion strength for tile adhesive. For curing condition factor, the maximum of percentage error between measured and predicted are referred to ± 36.5%, open time 20 minute, ± 33.5%, heat curing, ± 28.5%wet curing, and ± 16.0% dry curing respectively.

From the result, the plots in Figures 6 showed that a good relationship between experimented tensile adhesion strength and the predicted tensile adhesion strength could be obtained.

The results of the comparison of measured and predicted tensile adhesion strength at different curing condition in Figures 5 showed that the range of tensile adhesion strength between 0.180 and 1.310 MPa, the prediction yielded a percentage of error within about $\pm$36.50%. However, in Figure 6 showed that the range of %water demand between 0.201 and 0.350%, it was found that the maximum percentage of error was within $\pm$ 3.00%. It shows that the statistical mixture design technique has potential for tensile adhesion strength with different curing conditions at 28 days.

6. Conclusion

Using the mixture design with boundary restriction on the contents, it is possible to predict the tensile adhesion strength at different curing condition of tile adhesive and %water demand made with four blends ordinary Portland cement, sand, redispersible polymer powder, and cellulose ether. Based on the results of a minimum of seventeen design points, the prediction equations model can be established. For each formula, the prediction using a formula derived from the relationship between the predicted values with experimental value is within 36.5% of experimentally measured values. This was supported by the comparison of the calculated and tested tensile adhesion strengths.

Acknowledge

The authors would like to express the deepest gratitude to Asian Institute of Technology, Thailand for supporting the authors technically during this study. This work was supported by a master’s degree from Royal Thai Government Fellowship and Royal Irrigation Department.

Reference

[1] Bayer, R., lutz H. (2003). Dry mortar, Ullmann’s Encyclopedia of Industrial Chemistry, Wiley-VCH.

[2] Wang, D., Chen, Z., 1997. On predicting compressive strengths of mortars with ternary blends of cement, ggbfs and fly ash. Cement and Concrete Research, Vol. 27. No. 4, 487-493.

[3] Jian, T.D. , Pei-Yu, Y., Shu-Lin, L., Jin-Quan, Z.,1999. Extreme vertices design of concrete with combined mineral admixtures. Cement and Concrete Research 29, 957–960.

[4] Tiago, M., Telma, M.K, Maria Val´eria, R. V., Maria Elena, S.T., Vladi, O.C., 2006. Optimization of poorly compactable drug tablets manufactured by direct compression using the mixture experimental design. International Journal of Pharmaceutics 322, 87–95.

[5] Omer, Z., Sukru, K., 2006.Optimization of emulsion characteristics of beef, chicken and turkey meat mixtures in model system using mixture design. Meat Science 73 ,611–618.

[6] Zhou Jian, Z., Liu Xiao, L., Huang K.H., Dong, M.S., and Jiang, H.H., 2007. Application of the Mixture Design to Design the Formulation of Pure Cultures in Tibetan kefir. Agricultural Sciences in China, 6(11): 1383-1389.

[7] Phuriwat, L., Angkana, L., and Jakkapan, S., 2004. Optimization of Direct Compression Aspirin Tablet Using Statistical Mixture Design. CMU. Journal ,Vol. 3(2).

[8] Ademola, D.A., 2008. Mixture Experiments and their Applications in Welding Flux Design J. of the Braz. Soc. of Mech. Sci. & Eng. October-December 2008, No. 4 : 319.

[9] Mclean, R.A., Anderson, V.L., 1966. Extreme Vertices Design of Mixture Experiments, Technometrics, Vol. 8: 447-456.

[10] Design-Expert. Stat-Ease, Version 7.1.6 Trial, Minneapolis.

[11] Myers, R.H., Montgomery D.C., 1995. Response Surface Methodology: Process and Product Optimization Using Designed Experiments, Second Edition, New York: Wiley&Sons.

[12] EN1346, Adhesives for tile-Determination of open time, European Standard.

[13] EN1348, Adhesives for tile-Determination of tensile adhesion strength for cementitious adhesives, European Standard.

[14] EN12004, Adhesives for tile-Definitions and specification, European Standard.