Reuse Of Spent Catalyst From Sohar Refinery As An Additive In Cement Tiles

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Abstract

In Oman, two types of spent catalysts from refineries classified as waste materials are the reduced fluid cracking catalyst (RFCC) and spent alumina catalyst (SAC). In this study the RFCC spent catalyst from Sohar Refinery has been used to investigate its beneficial use as an additive in making cement tiles. Various percentage of spent catalyst (0%, 4%,6%,8%.10% and 12%) were prepared with Ordinary Portland Cement (OPC)which is an important raw material in the manufacturing mixture of tiles. In all prepared mixture the spent catalyst RFCC was substituted as an additive. Such mixtures were tested for physical, chemical and mechanical properties as well as durability and integrity.

Laboratory results indicated that the spent catalyst could find use as an additive in cement tiles.In general, the results showed that using spent catalyst as an additive gave results that fall within the required standard specification for OPC (CEM, EN 197-1:2000), (BS 4550., 1978) in terms of chemical composition, physical and mechanical properties. These results demonstrated that the spent catalyst RFCC could be added to cement tiles. This could potentially solve the environmental problem of disposal of spent catalyst from refineries as well as reduce the cost of cement tiles.

1. Introduction

Spent catalyst is generated as a waste material around the world from the cracking of petroleum in oil refineries. The disposal of spent catalyst is of major concern to oil refineries. In Oman, there are two types of spent catalysts generated by refineries: RFCC and SAC [6,12].

RFCC is used in the petroleum refinery cracking unit which is injected with oil feed to crack the trace oil and produce lighter oil such as gasoline, kerosene and naphthalene.

Approximately 20-25 tons per day of spent catalyst is generated at Sohar Refinery and the material produced is essentially in a fine powder form. In past years, the spent catalyst was sent to dumping sites or landfills. Although RFCC catalyst is relatively chemically stable, their catalytic life-time and efficiency decline with time. This is due to chemical degradation of the catalyst by reaction of oil feed with the catalyst phase causing its destruction and loss of reactivity [1].

It is also common that the catalyst active sites are irreversibly covered by chemical impurities, causing a permanent reduction of catalytic activity and requiring the catalyst to be replaced by a new one. However, with increasing environmental restriction and concerns, other waste management techniques should be explored. This could include recycling the spent catalyst in construction or conversion of the material into useful product in the manufacturing of cement, cement tiles or as an adsorbent in treating waste water. A lot of research has been done to explore the possibilities of different waste materials as additive in cement mixture [2,3,4,5,7,8,9,10,11,13]. This work explores the possibility of using RFCC from Sohar Refinery as an additive in making cement tiles.

2. Methodology and Experimentation

Spent catalyst RFCC was collected from Sohar Refinery weighing 7 Kg. The RFCC looks like sand so it was grinded to a powder and analyzed for its composition. The OPC was collected from Oman Cement Company weighing 20 Kg and analyzed for its composition. The spent catalyst and OPC were used to prepare the mixture to be used for the bottom layer of the cement tiles.

2.1. Chemical Test of Mixture

Six samples with different percentage of spent catalyst (RFCC) namely 0%, 4%, 6%, 8%, 10% and 12% were added with 450g of OPC to perform the chemical test. The chemical composition of mixture is shown in Table 1.

Table 1. Chemical Composition of the Tile Mixture

Component	Percentage (%)
S_iO_2	23.65
Al_2O_3	4.67
Fe_2O_3	4.34
CaO	60.58
MgO	1.62
SO_3	2.25
IR	0.63
LOI	2.26

2.2. Procedure for Manufacturing the Cement Tiles (Dual Layer)

Raw Materials: Cement tiles which are normally manufactured have dual layer namely wet layer and dry layer. The wet layer consists of white cement, potable water, marble powder and natural stone or aggregate or marble chips. The dry layer consists of sand and cement. In the present work the composition of dry layer is varied from that of normal by adding spent catalyst along with the normal composition.

Mix Proportion: The raw materials of the wet layer were mixed in the proportion of 1:3:4, i.e. 1 part of cement, 3 parts of marble powder and 4 parts of natural stone or aggregate with potable water to form a paste. The dry layer was mixed in a proportion of 1:6 i.e. 1 part of ordinary cement and 6 parts of washed sand.

Formation of Tile: The wet layer was poured into the desired size of mold (25x25cms mold) at a thickness of minimum 6-12mm and rest of the thickness of the mold was filled with dry layer; it was later pressed hydraulically at 150 - 180 bar.

Curing: The next process was curing where in the tiles were immersed in water for a minimum period of 24-36 hours. Then the tiles were taken out from the drum and dried in the sun for 3 days.

Polishing: After curing the tiles were left for polishing.

Drying: After polishing the tiles were left for drying for a period of 36 hours (the wet layer or top layer) and the tiles were ready. Figure 1 shows the picture of the ready tiles.

Figure 1. Cement Tiles

2.3. Tile Mixture Test

The tile mixture samples were now put to various tests to measure their quality.All samples were tested according to British Standard: CEM, EN 197-1:2000.The compressive strength test was determine by using 70.7 mm mortar cubes, made with specified sand, mixed and compacted by means of a standard vibration machine.Setting time testdeterminedthe initial and final setting time by using the setting time device. Expansion test was carried to determine the expansion of the mixture.

2.4. Product Tile Tests

The produced tile samples were now put to Water adsorption and Transverse tests to measure the quality of the tiles produced. All samples were tested according to British Standards: BS 4131:1973 and BS 4131., 1973. Water absorption by tile- face and total absorption of water test serves to determine the water absorption per unit area (absorption capacity) under atmospheric pressure and the dry bulk density. Transverse strength test used for determine the breaking load that to find from it the transverse strength of tiles by using specification formula.

3. Results and Analysis

3.1. Compressive Strength Test

The results were obtained for the compressive strength testing after 1, 2 and 28 days of casting the six samples consisting of Ordinary Portland Cement and Spent catalyst (RFCC): 0%, 4%, 6%, 8%, 10%, and 12%. The results of the samples gave good values for all percentages. According to (British Standard), CEM,EN 197-1:2000 the minimum value of compressive strength curing 1 day is $\geq$ 6 N/mm^2 , 2 days $\geq$ 10 N/mm^2 and 28 days $\geq$42 N/mm^2. Figure 2 shows the graphical comparison of the compressive strength tests while the respective values are provided in Table 2.

Table 2. Compressive strength test results

Spent Catalyst (%)	Compressive strength (N/mm^2)		
	1 day	2 day	28 day
0	6.25	15.21	45.90
4	9.80	20.00	49.70
6	10.28	19.57	52.20
8	7.50	17.72	50.60
10	6.28	14.68	47.20
12	6.00	14.37	47.70

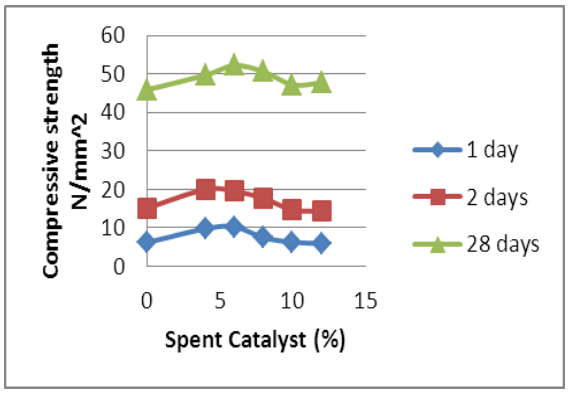

Figure 2. Compressive Strength Test

3.2. Setting Time Test

The water demand and setting time was determined by the setting time device.The water demand is the quantity of water which is needed to prepare the cement paste of standard consistency according to the British Standard method for Portland testing CEM, EN 197-1:2000. The water demand of cement paste of all samples has been found to increase which may be attributed to factors such as the specific surface area. Figure 3 shows the proportional relationship between depth (mm) and time for Sample 6.Similar relation is observed for the rest of the samples as well. The initial and final settingtime of all samples is between 3:30 hr to 4:00 hr which is a good value as according to standards the maximum value of setting time is 10:00 hr.

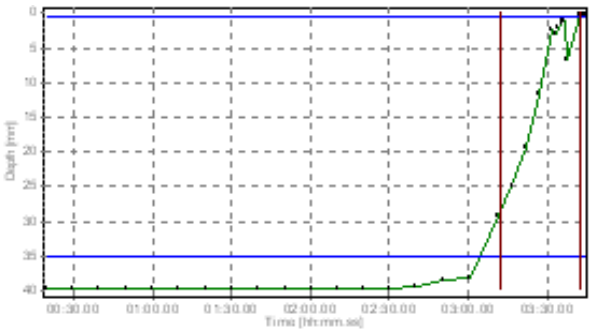

Figure 3 : Setting Time of Sample 6

Table 3: Setting time test results

Sample	Lower critical limit (LCL)		Upper critical limit (UCL)	
	Depth (mm)	Begin time min:sec	Depth (mm)	Begin time min:sec
1	35.0	178	0.5	231
2	35.0	179	0.5	234
3	35.0	205	0.5	231
4	35.0	195	0.5	233
5	35.0	191	0.5	219
6	35.0	192	0.5	223

3.3.Expansion Test

The expansion can be found out using the expansion formula, i.e., final diameter of sample – initial diameter of sample. The expansion result of samples was between 1mm-2mm which was again a good value as the maximum standard value of expansion is 10 mm.

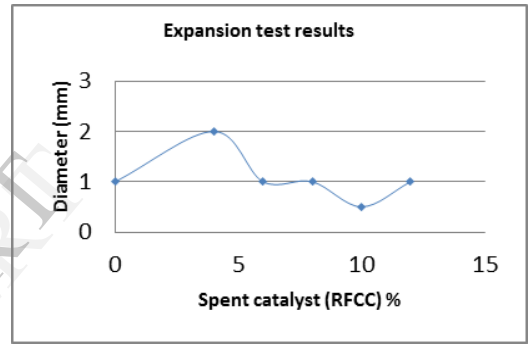

Figure 4. Spent Catalyst and Diameter

3.5. Transverse Strength Test

The results obtained for the transverse strength test for the different % of spent catalyst (RFCC): 0%, 4%, 6%, 8%, 10% and 12%. The results of the samples gave good values for all percentages. According to British standard, BS 4131:1973 the minimum value of transverse strength should be 3 N/mm^2.

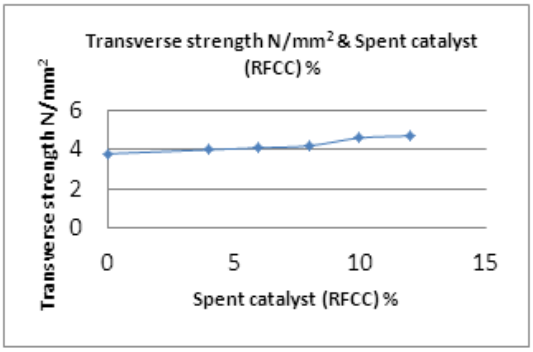

Figure 5. Transverse Strength N/mm^2& Spent catalyst (RFCC) %

3.6. Water Absorption by Tile- face

The water absorption by tile – face test measures the quantity of water absorbed in the face of cement tiles. The results of all cement tiles samples are got good values. According to British standard), BS 4131:1973 the water absorption by tile – face should be not more than 0.4 g/cm^2.

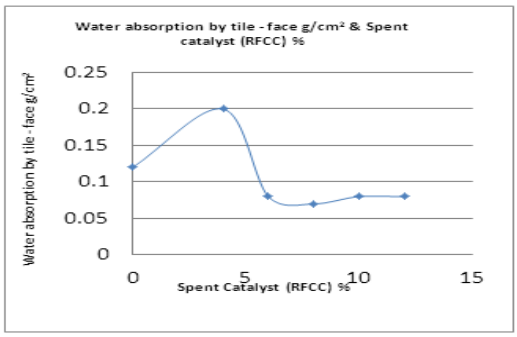

Figure 6. Water absorption by tile – face g/cm^2& Spent Catalyst (RFCC) %

3.7. Total Absorption of Water

The results of total absorption of water of all samples gave good values. Table 4shows the results for Sample 6 only and Figure7 shows the relationship between total water absorption and % of spent catalyst (RFCC). For other samples also the values obtained were in acceptable range. The value of water absorption should be not more than 8 % according to the British Standard, BS 4131:1973.

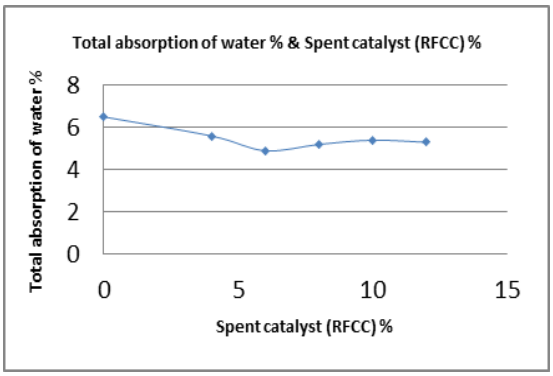

Figure 7: Total absorption of water % & spent catalyst (RFCC) %

Table 4. Cement Tiles Test result for Sample 6

Test Results					
A. Transverse Strength					
Client Sample No.	Span, mm	Width, mm	Thickness, mm	Breaking Load (N)	Transverse Strength (N/mm^2)
6-3	170	250	32	4700	4.7
6-4	170	250	33	4100	3.8
B. Water Absorption by Tile Face					
Client Sample No.	Dry Weight of Tile, g	Weight of Tile after Absorption of Water by Face, g	Weight of Water Absorbed by Tile-Face,g/cm^2		
6-1	4580	4630	0.08		
6-2	4594	4645	0.08		
C. Total Absorption of Water					
Client Sample No.	Dry Weight of Tile, g	Weight of Tile after Total Absorption of Water by Face, g	Total Absorption of Water,%		
6-3	4299	4562	6.1		
6-4	4446	4682	5.3		

4. Conclusion

Chemical and physical analysis carried out on the samples prepared from OPC and different percentages of spent catalyst (RFCC) show that these samples meet the normal standards and specification for Portland Cement Testing.

The compressive strength results of the samples gave good values for all percentages. According to (British Standard) , CEM,EN 197-1:2000 the minimum value of compressive strength curing 1 day is $\geq$ 6 N/mm^2 , 2 days $\geq$ 10 N/mm^2 and 28 days $\geq$ 42 N/mm^2 . In setting time results the initial and final time of all samples are between 3:30 hr to 4:00 hr which is a good value as according to standards the maximum value of setting time is 10:00 hr. The expansion results of samples is between 1mm-2mm which is again a good value when compared with the standard the maximum value of expansion that is 10 mm. According to the British Standard method BS-1881, all cement tiles test got very good results. In transverse strength test the results should be no single result shall be less than 3 N/mm^2, all samples got good values between 3.6 to 4.7 N/mm^2. In other hand the total absorption of water test and water absorption by tile – face are got good results , there are less than 8 % of total absorption and less than 0.4 g/cm^2 for water absorption by tile – face.

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