Orthogonal interactions and synergistic effects of salt treatment and calcination on rutile titanium dioxide pigment preparation

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Rutile titanium dioxide pigments were prepared by using metatitanic acid as raw material hydrolyzed from industrial low concentration titanyl sulfate solution via short sulfate process. The orthogonal interactions of the addition of salt treatment agent (ZnO , K_2O , P_2O_5) and the highest calcination temperature were investigated by using four-factor quadric orthogonal regression test. The metatitanic acid and rutile TiO_2 were characterized by XRD, particle size distribution and pigment properties test. The different salt addition and the highest calcination temperature had great effects on the achromatic power (TCS) of the rutile TiO_2 pigments. The orthogonal interactions regression model to TCS was built and showed highly repeatability and stability, which could be used to forecast the pigment properties and preset the salt treatment and calcination conditions. And the optimal salt treatment conditions were ZnO of 0.36%, K_2O of 0.70%, P_2O_5 of 0.20%, and the highest calcination temperature of 850 $^\circ\text{C}$.

Fig.1 XRD pattern for metatitanic acid

Fig.2 XRD patterns for rutile TiO_2 pigment

Table 3 Four-factor quadric orthogonal regression form and orthogonal regression table for TCS

No	Z_1 (A)	Z_2 (B)	Z_3 (C)	Z_4 (D)	Z_1Z_2 (AB)	Z_1Z_3 (AC)	Z_2Z_3 (BC)	Z_1Z_4 (AD)	Z_2Z_4 (BD)	Z_3Z_4 (CD)	Z_1' (A')	Z_2' (B')	Z_3' (C')	Z_4' (D')	Y (TCS)
1	1	1	1	1	1	1	1	1	1	1	0.257	0.257	0.257	0.257	1605
2	1	1	1	-1	1	1	1	-1	-1	-1	0.257	0.257	0.257	0.257	1599
3	1	1	-1	1	1	-1	-1	1	1	-1	0.257	0.257	0.257	0.257	1676
4	1	1	-1	-1	1	-1	-1	-1	-1	1	0.257	0.257	0.257	0.257	1612
5	1	-1	1	1	-1	1	-1	1	-1	1	0.257	0.257	0.257	0.257	1559
6	1	-1	1	-1	-1	1	-1	-1	1	-1	0.257	0.257	0.257	0.257	1487
7	1	-1	-1	1	-1	-1	1	1	-1	-1	0.257	0.257	0.257	0.257	1676
8	1	-1	-1	-1	-1	-1	1	-1	1	1	0.257	0.257	0.257	0.257	1766
9	-1	1	1	1	-1	-1	1	-1	1	1	0.257	0.257	0.257	0.257	1477
10	-1	1	1	-1	-1	-1	1	1	-1	-1	0.257	0.257	0.257	0.257	1471
11	-1	1	-1	1	-1	1	-1	-1	1	-1	0.257	0.257	0.257	0.257	1471
12	-1	1	-1	-1	-1	1	-1	1	-1	1	0.257	0.257	0.257	0.257	1374
13	-1	-1	1	1	1	-1	-1	-1	-1	1	0.257	0.257	0.257	0.257	1590
14	-1	-1	1	-1	1	-1	-1	1	1	-1	0.257	0.257	0.257	0.257	1495
15	-1	-1	-1	1	1	1	1	-1	-1	-1	0.257	0.257	0.257	0.257	1582
16	-1	-1	-1	-1	1	1	1	1	1	1	0.257	0.257	0.257	0.257	1458
17	1.664	0	0	0	0	0	0	0	0	0	2.026	-0.743	-0.743	-0.743	1678
18	-1.664	0	0	0	0	0	0	0	0	0	2.026	-0.743	-0.743	-0.743	1505
19	0	1.664	0	0	0	0	0	0	0	0	-0.743	2.026	-0.743	-0.743	1472
20	0	-1.664	0	0	0	0	0	0	0	0	-0.743	2.026	-0.743	-0.743	1668
21	0	0	1.664	0	0	0	0	0	0	0	-0.743	-0.743	2.026	-0.743	1463
22	0	0	-1.664	0	0	0	0	0	0	0	-0.743	-0.743	2.026	-0.743	1634
23	0	0	0	1.664	0	0	0	0	0	0	-0.743	-0.743	-0.743	2.026	1620
24	0	0	0	-1.664	0	0	0	0	0	0	-0.743	-0.743	-0.743	2.026	1562
25	0	0	0	0	0	0	0	0	0	0	-0.743	-0.743	-0.743	-0.743	1664
26	0	0	0	0	0	0	0	0	0	0	-0.743	-0.743	-0.743	-0.743	1667
27	0	0	0	0	0	0	0	0	0	0	-0.743	-0.743	-0.743	-0.743	1623
28	0	0	0	0	0	0	0	0	0	0	-0.743	-0.743	-0.743	-0.743	1718
29	0	0	0	0	0	0	0	0	0	0	-0.743	-0.743	-0.743	-0.743	1709

Table 6 Rutile content, crystal grain size, pigment properties and particle size distribution for rutile TiO_2 pigment

Rutile TiO_2	$X_R(\%)$	$L_{(110)}(nm)$	TCS	SCX	$Jasn$	D_{AV} nm	Pdi
8	99.3	115.6	1766	3.56	94.54	281	0.152
28	98.9	114.7	1718	3.34	94.63	273	0.149
30	98.8	113.5	1709	3.21	95.58	268	0.137
31	98.8	112.9	1712	3.09	94.60	270	0.161
32	98.7	112.4	1705	3.02	94.55	277	0.156
R930	/	/	1910	3.12	94.52	/	/

Sample 28#

Sample 30#

Fig.3 SEM photographs for rutile TiO_2 pigments