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Supplementary materials for:

Synthesis of Ti(SO₄)O solid acid nano-catalyst and its application for biodiesel production from used cooking oil

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Key words

Titanium sulphate oxide or titanium oxysulfate; solid acid catalyst; esterification & transesterification process; used cooking oil; nano-catalyst; and biodiesel.

Table S1 Characterisation of UCO feedstock

Parameters	Units	UCO
Flash point	°C	289
Kinematic viscosity at 40°C	mm ² /s	32. 91
Water content	vol.%	0.14
Density at 15°C	Kg/m ³	921
Acid value	mgKOH/g	4.04
Free fatty acid	mass%	2.034

Table S2 Comparison of de-convoluted O 1s of nano-sized TiO₂ with the reported values from the literature

References	Ti=O (eV)	C=O (eV)	O-H (eV)
Present work	530.21	531.22	532.52
Sanjines, et. al [1]	530.10	531.05	532.30
Liu, et al [2]	530.10	531.80	532.40
Ahmed, et al [3]	530.70	531.90	532.70

References:

1. Sanjines, R., H. Tang, H. Berger, F. Gozzo, G. Margaritondo, and F. Levy, Electronic structure of anatase TiO₂ oxide. *Journal of Applied Physics*, **1994**. 75(6): p. 2945-2951.
2. Liu, H., W. Yang, Y. Ma, Y. Cao, J. Yao, J. Zhang, and T. Hu, Synthesis and characterization of titania prepared by using a photoassisted sol-gel method. *Langmuir*, **2003**. 19(7): p. 3001-3005.
3. Ahmed, M.H., T.E. Keyes, and J.A. Byrne, The photocatalytic inactivation effect of Ag-TiO₂ on β -amyloid peptide (1-42). *Journal of Photochemistry and Photobiology A: Chemistry*, **2013**. 254: p. 1-11.

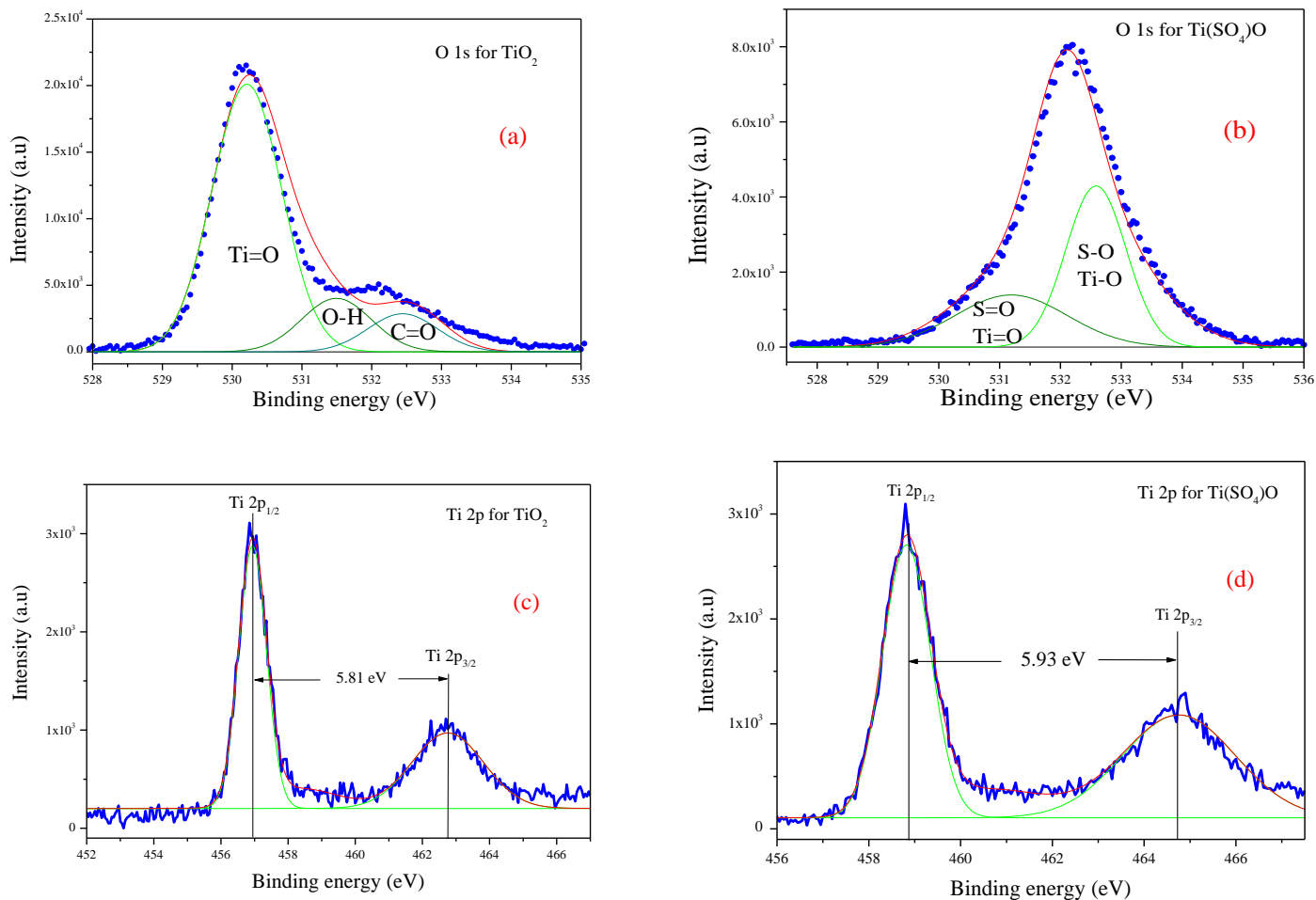


Figure S1: XPS Spectra showing the O 1s chemical environments for TiO_2 (a) and $\text{Ti}(\text{SO}_4)_2\text{O}$ nano-catalysts (b), the de-convolution of Ti 2p peaks for TiO_2 (c) and $\text{Ti}(\text{SO}_4)_2\text{O}$ nano-catalysts (d).

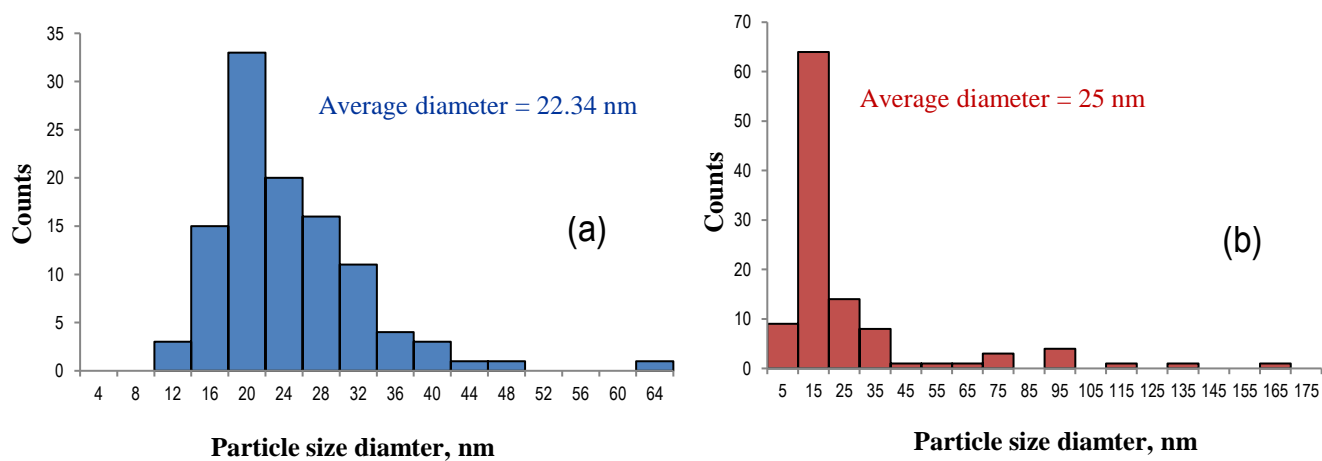


Figure S2: Particle size distributions of (a) TiO_2 and (b) $\text{Ti}(\text{SO}_4)_2\text{O}$ nano-catalyst samples

Table S3 Comparison of d-spacing values from this experiment with the reported d-spacing values given in the JCPDS- ICDD reference cards

d-Spacing, Å	h k l	101	103	004	112	200	105	211	213	204
	JCPDS-ICDD	3.5165	2.4305	2.3781	2.3323	1.8923	1.6998	1.6664	1.4931	1.4807
TiO ₂ , Anatase	XRD	3.5168	2.4306	2.3780	2.3324	1.8925	1.6998	1.6665	1.4932	1.4808
	SAED	3.5476	---	2.3754	---	1.8240	---	1.6612	---	---
d-Spacing, Å	h k l	110	101	200	111	210	211	220	002	310
	JCPDS-ICDD	3.2488	2.4862	2.2972	2.1866	2.0547	1.6873	1.6244	1.4782	1.4529
TiO ₂ , Rutile	XRD	3.2485	2.4876	2.2970	2.1875	2.0545	1.6876	1.6242	1.4795	1.4528
	SAED	3.1815	---	2.2870	---	2.0997	---	1.6185	---	---
d-Spacing, Å	h k l	200	201	111	301	112	410	122	222	611
	JCPDS-ICDD	5.4743	4.1669	3.7716	3.1735	2.6447	2.4170	1.9758	1.8859	1.6616
Ti(SO ₄)O	XRD	5.4751	4.1673	3.7714	3.1738	2.6447	2.4172	1.9757	1.8858	1.6618
	SAED	5.4795	4.1639	3.7738	3.1672	2.6317	2.4175	1.9639	1.8934	1.6656

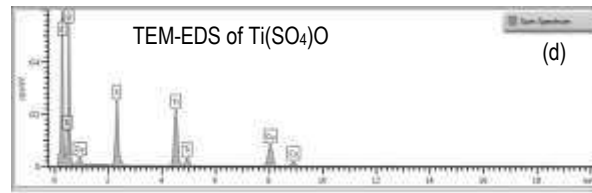
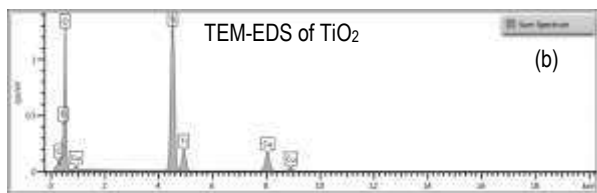
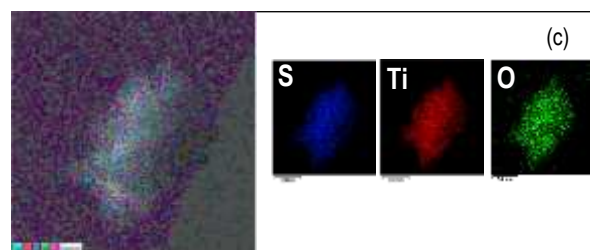
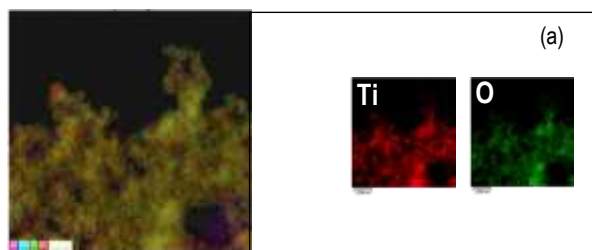


Figure S3: TEM mapping for (a) TiO₂ and (c) Ti(SO₄)O nano-catalysts and energy dispersive X-ray spectroscopy for (b) TiO₂ and (d) Ti(SO₄)O nano-catalysts.