Facile hydrothermal synthesis of crystalline Ta_2O_5 nanorods, $MTaO_3$ (M=H, Na, K, Rb) nanoparticles, and their photocatalytic behaviour.

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Supporting Information

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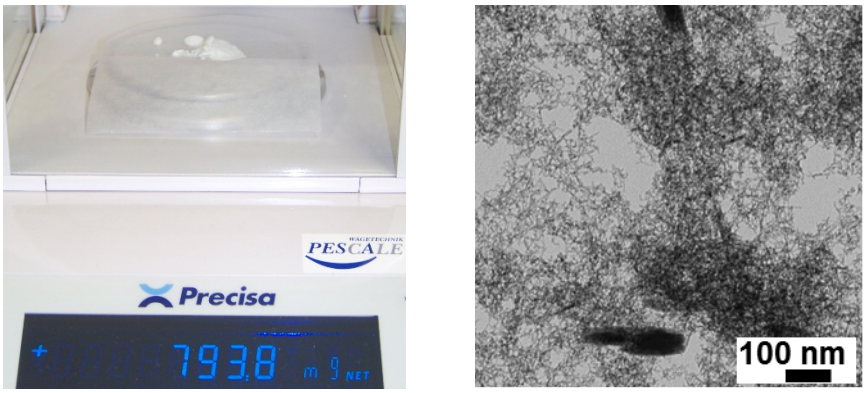

Fig. S1. Left: Photograph of the product obtained by an upscaled reaction. Right: Reaction TEM image of a snapshot after 12 h for pH = 4.

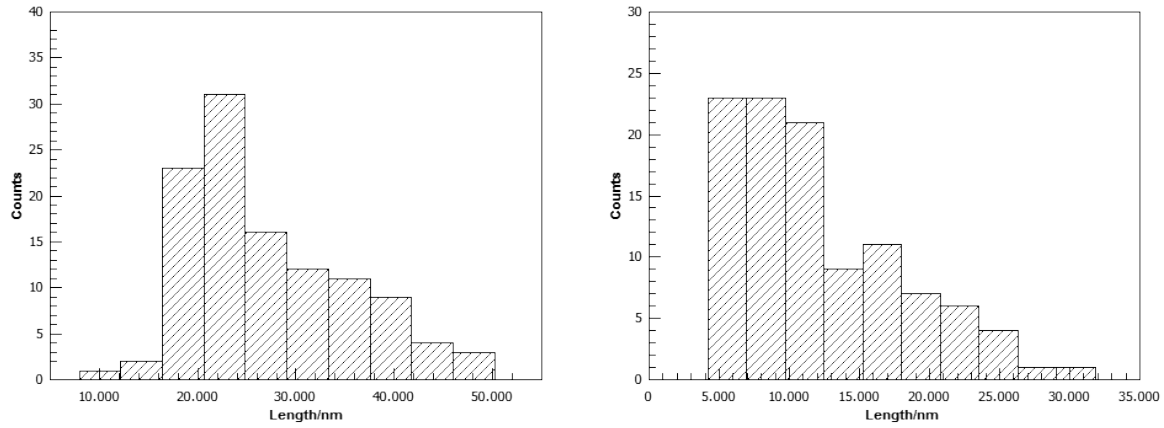

Fig. S2. Histogramm of the $NTaO_3$ nanorods synthesized at pH=12 (left) and pH=13 (right).

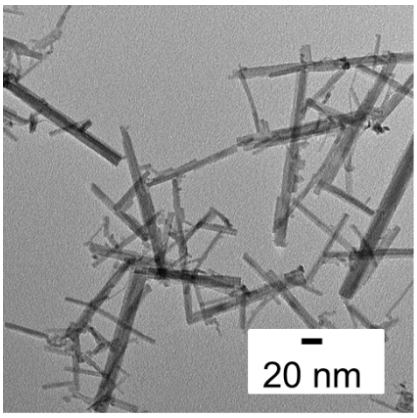

Fig. S3. TEM micrograph of the Ta_2O_5 nanorods synthesized with 5 mM NaOH.

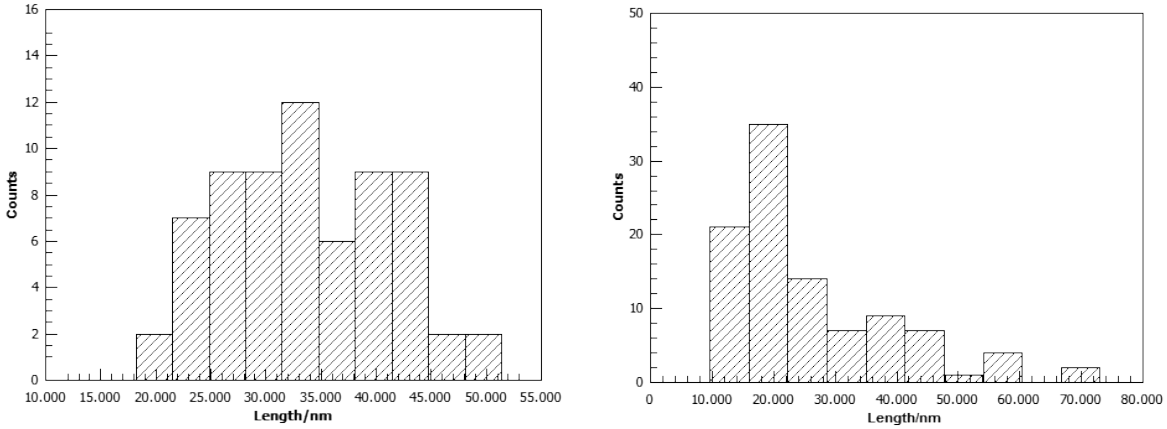

Fig. S4. Histograms of the $KTaO_3$ (left) and $RbTaO_3$ (right) nanoparticles synthesized at pH=12.

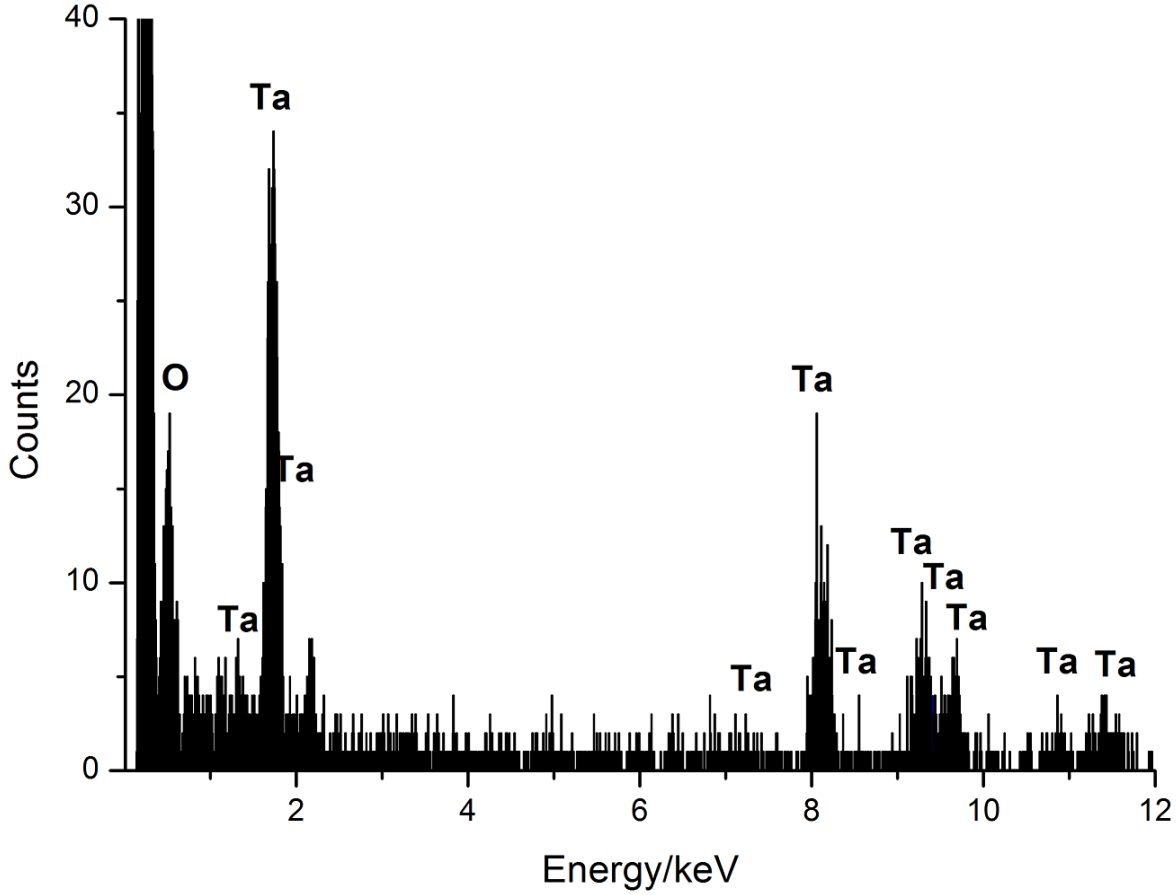

Fig. S5. EDX of the rods with 5 mM RbOH.

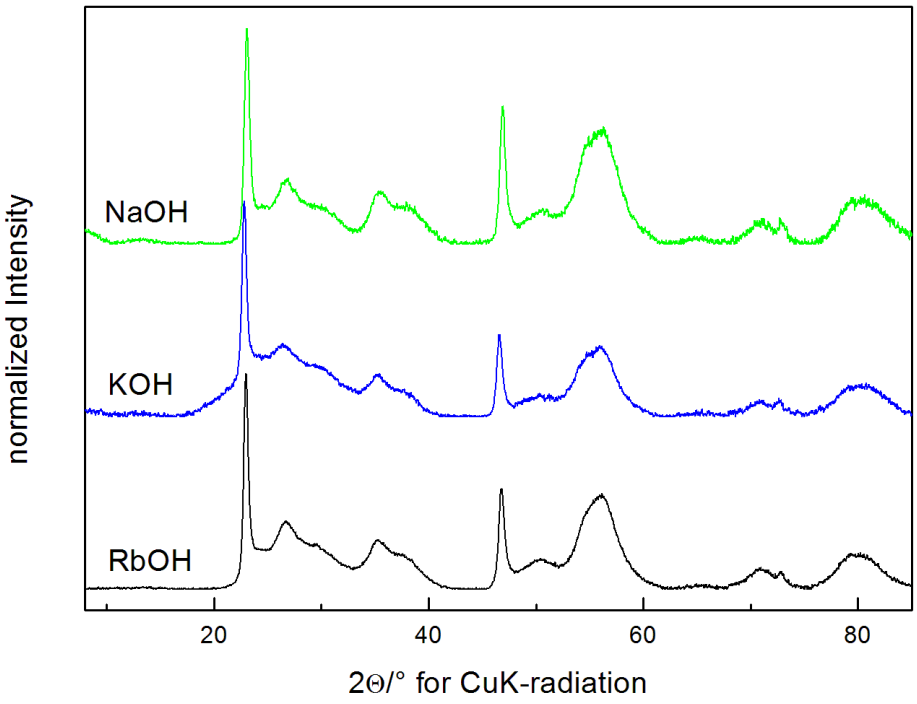

Fig. S6. X-ray diffraction patterns of the tantalum oxide rods with 5 mM NaOH, KOH and RbOH respectively. Reflection profiles look wider than those in Fig. 2 as these were recorded on diffractometers with different instrumental broadening.

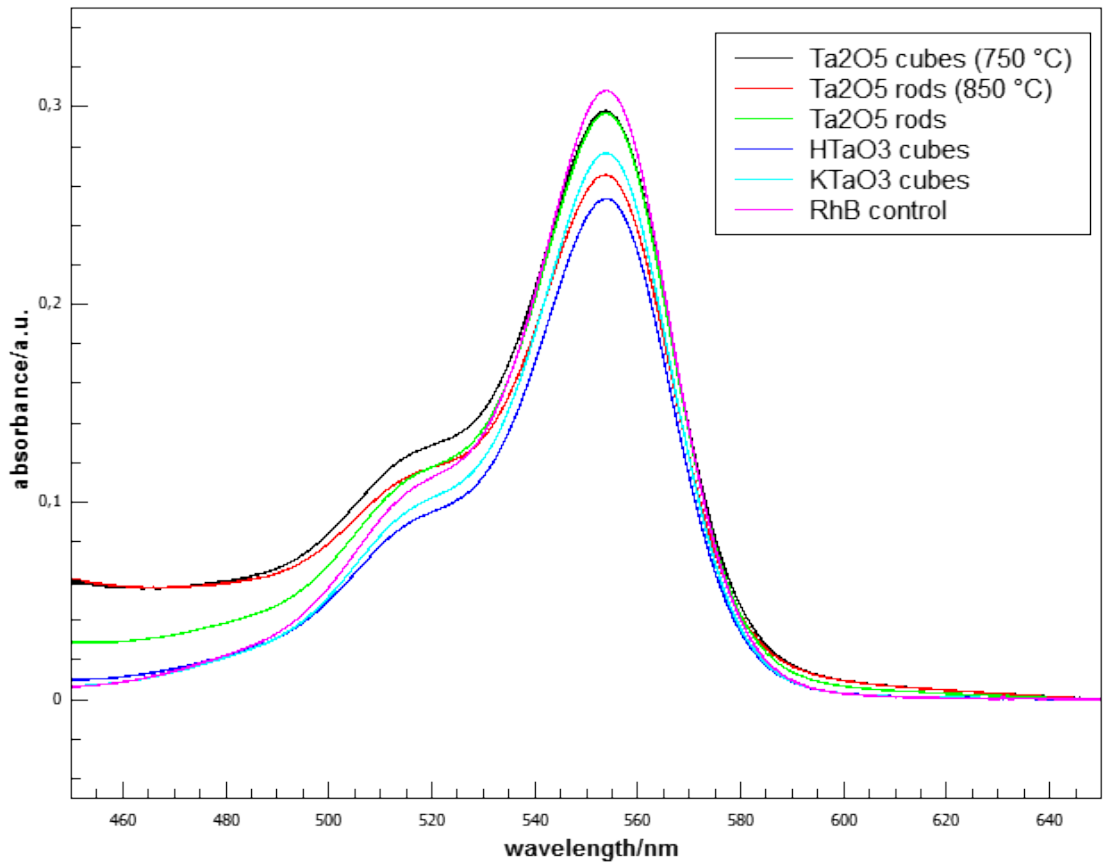

Fig. S7. UV-VIS spectra of the samples after stirring in the dark for 18 minutes and centrifugation of the nanoparticles.

Tab. S1. Measurement and refinement parameters of the x-ray diffraction pattern of products prepared at different pH-values

	pH = 3	pH = 4	pH = 7	pH = 8	pH = 9	pH = 12	pH = 13
Diffractometer	Siemens D5000						
Sample preparation	Fine powder fixed between two stripes of Scotch™ tape						
Measuring mode	Transmission						
Wavelength	1.540596						
Measuring range	$10 \leq 2\Theta\ /° \leq 90;\ 0.71 \leq Q\ /Å^{-1} \leq 5.77$						
Temperature /K	298K						
Profile Fit	Rietveld refinement according to reported crystal structure models						
Background	Chebyshev						
Profile function	Fundamental Parameters Approach						
Program	TOPAS Academic V5						
Total No. of Parameters / Background	29 / 20	29 / 20			33 / 20	27 / 20	33 / 20
R_{exp}	1.97	3.47			2.06	1.96	1.94
R_{wp}	4.21	6.55			6.72	5.17	4.97
GoF	2.92	1.89			3.26	2.64	2.52
DW	0.36	0.69			0.25	0.30	0.52
Ta_2O_5 – *oP*14							
Space group	*Pccm*						
Cell parameters /Å	a = 3.6188(5) b = 6.304(2) c = 7.7910(1)	a = 3.6264(5) b = 6.318(2) c = 7.8097(9)			a = 3.5810(1) b = 6.14(4) c = 7.775(2)		
Crystallite size / nm	41(1) (ab) 70(1) (c)	47(1) (ab) 75(1) (c)			48(2)		
Fraction /%wt	100	100			15(2)		
Biso	3.3(1)	3.4(1)			3.0 fixed		
Preferred Orientation	1.33(1) / (020)	1.38(1) / (020)			3.3(2) / (020)		
$H_2Ta_2O_6{\cdot}H_2O$ – *cF*104							
Space group	*Fd*-3*m*						
Cell parameter /Å					a = 10.457(2)	a = 10.5249(3)	a = 10.5237(5)
Crystallite size / nm					33(1)	47(1)	21(1)
Fraction /%wt					85(1)	100	83(1)
Biso					3 (fixed)	3 (fixed)	3 (fixed)
site occupation factor 16*d*					1.00(6)	0.42(1)	0.39(2)
Site occupation factor 8b					1.0(1)	0.37(3)	0.28(4)
approx. composition					$Na_2Ta_2O_6{\cdot}H_2O$	$Na_{0.84}H_{1.16}Ta_2O_6{\cdot}0.37H_2O$	$Na_{0.78}H_{1.22}Ta_2O_6{\cdot}0.28H_2O$
$NaTaO_3$ – *oP*10							
Space group							*Pnma*
Cell parameter /Å							a = 5.5258(3) b = 7.7971(4) c = 5.4839(3)
Crystallite size / nm							> 100nm
Fraction /%wt							17(2)
Biso							3.0 (fixed)

Tab. S2. Measurement and refinement parameters of the x-ray diffraction pattern of the cube-shaped nanoparticles with different bases.

	Na	K	Rb
Diffractometer	Siemens D5000		
Sample preparation	Fine powder fixed between two stripes of Scotch™ tape		
Measuring mode	Transmission		
Wavelength	1.540596		
Measuring range	$10 \leq 2\Theta$ /° ≤ 90; $0.71 \leq Q$ /Å$^{-1} \leq 5.77$		
Temperature /K	298K		
Profile Fit	Rietveld refinement according to reported crystal structure models		
Background	Chebyshev		
Profile function	Fundamental Parameters Approach		
Program	TOPAS Academic V5		
Total No. of Parameters / Background	27 / 20		
R_{exp}	1.96	1.97	3.59
R_{wp}	5.17	5.57	7.41
GoF	2.64	2.83	2.06
DW	0.30	0.30	0.49
$H_2Ta_2O_6 \cdot H_2O$ – *cF*104			
Space group	*Fd*-3*m*		
Cell parameter /Å	10.5249(3)	10.6324(2)	10.6146(7)
Crystallite size / nm	47(1)	68(1)	31(1)
Fraction /%wt	100	100	100
Biso	3 (fixed)		
site occupation factor 16*d*	0.42(1)	0.488(8)	0.098(5)
Site occupation factor 8b	0.37(3)	0.00(3)	1.00(5)
approx. composition	$Na_{0.88}H_{1.12}Ta_2O_6 \cdot 0.37H_2O$	$K_{0.97}H_{1.03}Ta_2O_6$	$Rb_{0.2}H_{1.8}Ta_2O_6 \cdot 1H_2O$

Tab. S3. XRF data of the cube-shaped $MTaO_3$ (M=Na, K, Rb) before and after treatment with 2 M HCl.

Nanoparticles	Analyte/ Compound Formula	Calibration Status	Measured /kcps	Used/kcps	Concentration/ %	Status
$NaTaO_3$	Na	Calibrated	6.102	5.587	12.551	Calculate
	Ta	Calibrated	77.874	78.487	87.449	Calculate
HCl washed $NaTaO_3$	Al	Calibrated	0.101	0.097	0.187	Calculate
	Ta	Calibrated	46.512	46.769	99.813	Calculate
$KTaO_3$	Al	Calibrated	0.137	0.124	0.102	Calculate
	Si	Calibrated	0.569	0.460	0.481	Calculate
	K	Calibrated	7.553	7.554	9.019	Calculate
	Ta	Calibrated	100.934	101.813	90.398	Calculate
HCl washed $KTaO_3$	Si	Calibrated	0.614	0.490	0.540	Calculate
	S	Calibrated	0.346	0.232	0.101	Calculate
	K	Calibrated	0.483	0.434	0.551	Calculate
	Ta	Calibrated	104.688	105.518	98.808	Calculate
$RbTaO_3$	Rb	Calibrated	151.266	151.206	27.107	Calculate
	Ta	Calibrated	84.145	84.646	72.893	Calculate
HCl washed $RbTaO_3$	Ta	Calibrated	84.973	85.650	100.000	Calculate

Tab. S4. Measurement and refinement parameters of the x-ray diffraction pattern of the acid treated cube-shaped nanoparticles and the heat treated rods and cube shaped nanoparticles

	Pristine	After HCl treatment	750°C	850°C
Diffractometer	Siemens D5000			
Sample preparation	Fine powder fixed between two stripes of Scotch™ tape			
Measuring mode	Transmission			
Wavelength	1.540596			
Measuring range	$10 \leq 2\Theta$ /° ≤ 90; $0.71 \leq Q$ /Å$^{-1} \leq 5.77$			
Temperature /K	298K			
Profile Fit	Rietveld refinement according to reported crystal structure models			
Background	Chebyshev			
Profile function	Fundamental Parameters Approach			
Program	TOPAS Academic V5			
Total No. of Parameters / Background	27 / 20	27 / 20	25 / 20	25 / 20
R_{exp}	1.97	1.98	2.03	2.12
R_{wp}	5.57	5.37	5.75	10.78
GoF	2.83	2.72	2.83	5.09
DW	0.30	0.31	0.33	0.16
$H_2Ta_2O_6 \cdot H_2O$ – *cF*104				
Space group	*Fd*-3*m*			
Cell parameter /Å	10.6324(2)	10.6014(2)		
Crystallite size / nm	68(1)	71(1)		
Fraction /%wt	100	100		
Biso	3 (fixed)			
site occupation factor 16*d*	0.488(8)	0.000(7)		
Site occupation factor 8b	0.00(3)	0.45(3)		
approx. composition	$K_{0.97}H_{1.03}Ta_2O_6$	$H_2Ta_2O_6 \cdot 0.45H_2O$		
Ta_2O_5 – *oP*14				
Space group			*Pccm*	
Cell parameters /Å			a = 3.6380(3), b = 6.2670(6), c = 7.7824(5)	a = 3.6565(4), b = 6.2108(8), c = 7.7758(8)
Crystallite size / nm			33(1) (ab) 49(1) (c)	20(1) (ab) 57(1) (c)
Fraction /%wt			100%	100%
Biso			3.09(6)	3.64(9)
Preferred Orientation			1.052(5) / (0 2 0)	1.086(6) / (0 2 0)