**A switchable-oxidative cellulose filter paper bearing immobilized Mn(III)-salen complex for alcohol oxidation**

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**Different synthetic routes to synthesis filter paper 4 (failed/ low efficiency routes)**



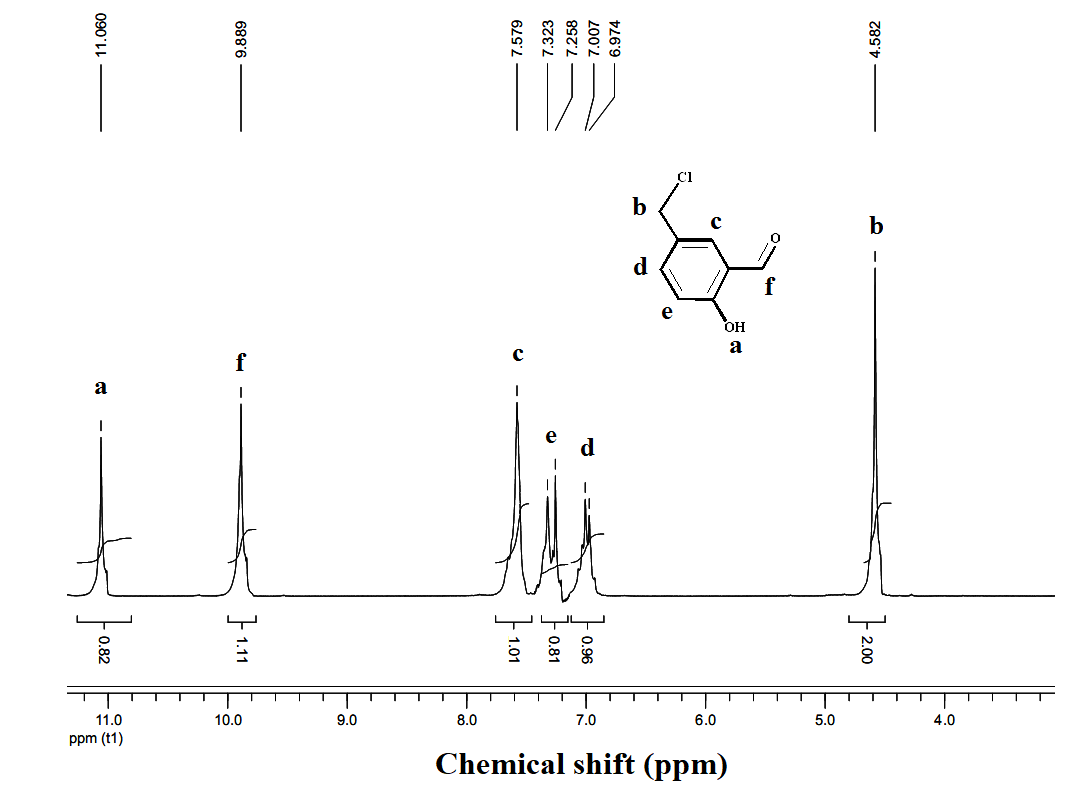
**Scheme S1** Different synthetic routes to synthesis filter paper **4**

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**Fig. S1** FTIR spectra of (a) salen ligand (**2**), (b) salen-TEMPO ligand (**3**), and (c) Mn(III)-salen-TEMPO complex (**4**)

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**Fig. S2** ATR-IR spectra of (a) plain (unmodified) cellulose filter paper, (b) SiCFP, and (c) FP@Si-MnIII-Salen-TEMPO



**Fig. S3** 1H-NMR spectrum of 5-chloromethyl salicylaldehyde (**1**) (CDCl3, 300 MHz) [1]

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**Fig. S4** 13C-NMR spectrum of 5-chloromethyl salicylaldehyde (**1**) (CDCl3, 75 MHz)

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**Fig. S5** 1H-NMR spectrum of salen ligand (**2**) (DMSO-*d6*, 300 MHz)

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**Fig. S6** 13C-NMR spectrum of salen ligand (**2**) (DMSO-*d6*, 75 MHz)

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**Fig. S7** 1H-NMR spectrum of Salen-TEMPO complex (**3**) (DMSO-*d6*, 300 MHz)

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**Fig. S8** 13C-NMR spectrum of Salen-TEMPO complex (**3**) (DMSO-*d6*, 75 MHz)

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**Fig. S9** 1H-NMR spectrum of Mn(III)-salen-TEMPO complex (**4**) (DMSO-*d6*, 300 MHz)

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**Fig. S10** 13C-NMR spectrum of Mn(III)-salen-TEMPO complex (**4**) (DMSO-*d6*, 75 MHz)

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**Fig. S11** (a) XRD pattern, (b) EDX spectrum, (c) XPS overal survey analysis, (d) TGA analysis, and (e) FESEM image of the pristine cellulose filter paper

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**Fig. S12** The 3D central design curves for foundation of optimization ranges for maximum response in the oxidation of benzyl alcohol to benzaldehyde

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**Fig. S13** The 2D central design curves for foundation of optimization ranges for maximum response in the oxidation of benzyl alcohol to benzaldehyde

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**Fig. S14** Accuracy of the predicted model *vs*. actual values

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**Fig. S15** (a) FESEM image and (b) EDX analysis (inset Table represents the elemental composition results in mean of 5 points) of FP@Si-MnIII-Salen-TEMPO after four consecutive recycles

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**Fig. S16** Influence of pH over Pd leaching of FP@Si-MnIII-Salen-TEMPO

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**Fig. S17** Swellability and shrinkage measurements of FP@Si-MnIII-Salen-TEMPO in (a) EtOH: H2O (2:1, v/v) and (b) mixture of NaOCl/ EtOH: H2O (2:1, v/v) for 5 consecutive wetting-drying cycles

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**Fig. S18** Swellability and shrinkage of the plain (unmodified) and silylated filter paper in EtOH: H2O solvent for 5 consecutive drying-wetting cycles

**Table S1** Elemental analyses of the silylated filter paper resulted from ICP as well as EDX analyses

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| Element | CFP | | SiCFP(aq)a | | SiCFP(vap)b | |
| ICP (wt%)c | EDXd (wt%) | ICPc (wt%) | EDXd (wt%) | ICPc (wt%) | EDXd (wt%) |
| C | - | 51.36 | - | 48.60 | - | 48.09 |
| O | - | 48.64 | - | 34.96 | - | 34.18 |
| Si | - | - | 14.68 | 11.36 | 12.90 | 11.96 |
| Cl | - | 0.00 | - | 5.08 | - | 5.77 |

a Cellulose filter paper silylated from aqueous solution of CPTES as a soaking method. b Cellulose filter paper silylated from chemical vapor deposition method for 6h time interval. c Resulted from ash of the silylated filter paper. d Mean of 5 points

**Table S2** Swelling measurements of the plain, silylated, and FP@Si-MnIII-Salen-TEMPO

filter papers

|  |  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| Solvent | | Swelling (g/mL) | | | | | | | | |
| H2O | H2O:EtOH | DMF | EtOH | MeOH | DMSO | Toluene | Acetic acid | Acetone |
| Filter paper | Plain | 8.8 | 11.2 | 13.3 | 6.8 | 7.5 | 14.2 | 0.6 | 5.9 | 1.5 |
| Silylated | 10.0 | 15.2 | 14.0 | 9.1 | 9.6 | 15.7 | 0.6 | 7.2 | 3.0 |
| Filter paper **7** | 9.4 | 14.6 | 13.5 | 8.4 | 9.0 | 14.0 | 0.4 | 6.1 | 2.0 |

**ANOVA for Quadratic model**

**Table S3** Fit Statistics of the resultsa

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| Std. Dev. | 2.71 |  | R² | 0.9876 |
| Mean | 70.83 |  | Adjusted R² | 0.9742 |
| C.V. % | 3.83 |  | Predicted R² | 0.9427 |
|  |  |  | Adeq Precision | 29.1957 |

a The **Predicted R²** of 0.9427 is in reasonable agreement with the **Adjusted R²** of 0.9742; i.e. the difference is less than 0.2.

**Adeq Precision** measures the signal to noise ratio. A ratio greater than 4 is desirable. Your ratio of 29.196 indicates an adequate signal. This model can be used to navigate the design space.

**Table S4** ANOVA for Response Surface Quadratic Model in model benzyl alcohol oxidation [analysis of variance table]a

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| Source | Sum of Squares | df | Mean Square | F-value | p-value |  |
| Block | 83.27 | 2 | 41.63 |  |  |  |
| Model | 7615.08 | 14 | 543.93 | 73.80 | < 0.0001 | significant |
| A-Mn loading | 1855.04 | 1 | 1855.04 | 251.68 | < 0.0001 |  |
| B-O2 | 0.0417 | 1 | 0.0417 | 0.0057 | 0.9412 |  |
| C-T | 360.38 | 1 | 360.38 | 48.89 | < 0.0001 |  |
| D-NOF | 2542.04 | 1 | 2542.04 | 344.89 | < 0.0001 |  |
| AB | 0.0625 | 1 | 0.0625 | 0.0085 | 0.9280 |  |
| AC | 85.56 | 1 | 85.56 | 11.61 | 0.0047 |  |
| AD | 138.06 | 1 | 138.06 | 18.73 | 0.0008 |  |
| BC | 7.56 | 1 | 7.56 | 1.03 | 0.3296 |  |
| BD | 45.56 | 1 | 45.56 | 6.18 | 0.0273 |  |
| CD | 7.56 | 1 | 7.56 | 1.03 | 0.3296 |  |
| A² | 18.57 | 1 | 18.57 | 2.52 | 0.1364 |  |
| B² | 0.8601 | 1 | 0.8601 | 0.1167 | 0.7381 |  |
| C² | 1732.65 | 1 | 1732.65 | 235.08 | < 0.0001 |  |
| D² | 1053.65 | 1 | 1053.65 | 142.95 | < 0.0001 |  |
| Residual | 95.82 | 13 | 7.37 |  |  |  |
| Lack of Fit | 83.82 | 10 | 8.38 | 2.10 | 0.2948 | not significant |
| Pure Error | 12.00 | 3 | 4.00 |  |  |  |
| Cor Total | 7794.17 | 29 |  |  |  |  |

a Factor coding is **Coded**. Sum of squares is **Type III - Partial**

The **Model F-value** of 73.80 implies the model is significant. There is only a 0.01% chance that an F-value this large could occur due to noise.

**P-values** less than 0.0500 indicate model terms are significant. In this case A, C, D, AC, AD, BD, C², D² are significant model terms. Values greater than 0.1000 indicate the model terms are not significant. If there are many insignificant model terms (not counting those required to support hierarchy), model reduction may improve your model.

The **Lack of Fit F-value** of 2.10 implies the Lack of Fit is not significant relative to the pure error. There is a 29.48% chance that a Lack of Fit F-value this large could occur due to noise. Non-significant lack of fit is good -- we want the model to fit.

### Table S5 Coefficients in Terms of Coded Factors

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| Factor | Coefficient Estimate | df | Standard Error | 95% CI Low | 95% CI High | VIF |
| Intercept | 82.67 | 1 | 1.11 | 80.27 | 85.06 |  |
| Block 1 | -2.23 | 2 |  |  |  |  |
| Block 2 | 0.4667 |  |  |  |  |  |
| Block 3 | 1.77 |  |  |  |  |  |
| A-Mn loading | 8.79 | 1 | 0.5542 | 7.59 | 9.99 | 1.0000 |
| B-O2 | -0.0417 | 1 | 0.5542 | -1.24 | 1.16 | 1.0000 |
| C-T | -3.88 | 1 | 0.5542 | -5.07 | -2.68 | 1.0000 |
| D-NOF | 10.29 | 1 | 0.5542 | 9.09 | 11.49 | 1.0000 |
| AB | 0.0625 | 1 | 0.6787 | -1.40 | 1.53 | 1.0000 |
| AC | -2.31 | 1 | 0.6787 | -3.78 | -0.8462 | 1.0000 |
| AD | 2.94 | 1 | 0.6787 | 1.47 | 4.40 | 1.0000 |
| BC | 0.6875 | 1 | 0.6787 | -0.7788 | 2.15 | 1.0000 |
| BD | 1.69 | 1 | 0.6787 | 0.2212 | 3.15 | 1.0000 |
| CD | -0.6875 | 1 | 0.6787 | -2.15 | 0.7788 | 1.0000 |
| A² | -0.8229 | 1 | 0.5184 | -1.94 | 0.2970 | 1.05 |
| B² | 0.1771 | 1 | 0.5184 | -0.9428 | 1.30 | 1.05 |
| C² | -7.95 | 1 | 0.5184 | -9.07 | -6.83 | 1.05 |
| D² | -6.20 | 1 | 0.5184 | -7.32 | -5.08 | 1.05 |

The coefficient estimate represents the expected change in response per unit change in factor value when all remaining factors are held constant. The intercept in an orthogonal design is the overall average response of all the runs. The coefficients are adjustments around that average based on the factor settings. When the factors are orthogonal the VIFs are 1; VIFs greater than 1 indicate multi-colinearity, the higher the VIF the more severe the correlation of factors. As a rough rule, VIFs less than 10 are tolerable.

**Table S6** Effect of different Mn loading amounts (on the catalytic paper **7)** over the benzyl alcohol oxidation of benzaldehydea

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| Entry | mmolb of Mn/95 cm2 | Time (min)/ cyclesb | Conversion (%)c | Selectivity (%) |
| 1 | 0.5 | 37/ 3 | 30 | 96 |
| 2 | 1.0 | 37/ 3 | 70 | 86 |
| 3 | 1.5 | 38/ 3 | 80 | 96 |
| 4 | 2.0 | 37/ 3 | 98 | 98 |
| 5 | 2.5 | 42/ 3 | 96 | 96 |

a Reaction conditions: Benzyl alcohol (1.0 mmol), H2O:EtOH (5.0 mL, 1:2, v/v), O2 bubbling (inside the reaction mixture on the filter paper, 1 ml/min), catalytic filter paper **7** (placed on a glass funnel), R.T.

b Based on ICP analysis. c Total time spent for 3 consecutive re-filtration of the residue. d Isolated yield.

**Table S7** Influence of oxidant on the oxidation of benzyl alcohol to benzaldehyde catalyzed by FP@Si-MnIII-Salen-TEMPO catalytic filter paper

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| Entry | Oxidant | Time (min)/ cyclesb | Conversion (%)c | Selectivity (%) |
| 1 | Air | 38/ 3 | 60 | 96 |
| 2 | H2O2 35% | 37/ 3 | 95 | 7 |
| 3 | Oxoned | 42/ 3 | 95 | 75 |
| 4 | O2e | 37/ 3 | 98 | 98 |
| 5 | *m*-CPBAf | 38/ 3 | 94 | 96 |
| 6 | TBHPg | 40/ 3 | 98 | 90 |
| 7 | NaIO4 | 38/ 3 | 72 | 5h |
| 8 | NaOCl | 37/ 3 | 98 | 3h |
| 9 | -i | 36/ 3 | 20 | 10 |

a Reaction conditions: Benzyl alcohol (1.0 mmol), H2O:EtOH (5.0 mL, 1:2, v/v), oxidant (2.5 mmol), catalytic filter paper (**7**, placed on a glass funnel containing 2.0 mmol Mn/95cm2), R.T. b Number of filtration. Total time spent for 3 consecutive re-filtration of the residue. c GC yield. d KHSO5. e Molecular oxygen was bubbled into the reaction mixture by immersing the syringe attached to the O2 source. f *meta*-Chloroperoxybenzoic acid. g *tert*-BuOOH. h Benzoic acid was selectively produced. i N2 gas was replaced by O2 gas and the reaction was performed under a sealed condition. In each cycle (filtration), N2 gas was passed through the flask to empty the flask from air.

**Table S8** Effect of O2 flow rate over the oxidation of benzyl alcohol to benzaldehyde catalyzed by FP@Si-MnIII-Salen-TEMPO catalytic filter papera

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| Entry | O2 flow rate (mL/min) | Time (min)/ cyclesb | Conversion (%)c | Selectivity (%) |
| 1 | Air | 38/ 3 | 60 | 96 |
| 2 | 0.5 | 38/ 3 | 88 | 96 |
| 3 | 1.0 | 38/ 3 | 98 | 96 |
| 4 | 2 | 38/ 3 | 93 | 92 |
| 5 | 2.5 | 38/ 3 | 95 | 88 |
| 6 | 5 | 38/ 3 | 92 | 78 |
| 7 | 10 | 38/ 3 | 90 | 67 |

a Reaction conditions: Benzyl alcohol (1.0 mmol), H2O:EtOH (5.0 mL, 1:2, v/v), O2 bubbling (inside the reaction mixture on the filter paper), catalytic filter paper **7** (placed on a glass funnel containing 2.0 mmol Mn/95cm2), R.T.

**Table S9** Effect of solvent on the oxidation of benzyl alcohol to benzaldehyde catalyzed by FP@Si-MnIII-Salen-TEMPO catalytic filter paper

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| Entry | Solvent | Time (min)/ cyclesb | Conversion (%)c | Selectivity (%) |
| 1 | Acetic acid | 40/ 3 | 98 | 98 |
| 2 | CH3CN | 32/ 3 | 60 | 70 |
| 3 | H2O | 37/ 3 | 70 | 88 |
| 4 | EtOH | 37/ 3 | 95 | 98 |
| 5 | H2O:EtOH (1:2, v/v) | 37/ 3 | 98 | 98 |
| 6 | THF | 30/ 3 | 40 | 90 |
| 7 | DCM | 30/ 3 | 40 | 90 |
| 8 | EtOAc | 35/ 3 | 55 | 75 |
| 9 | DMSO | 45/ 3 | 75 | 96 |

a Reaction conditions: Benzyl alcohol (1.0 mmol), solvent (5.0 mL), O2 bubbling (inside the reaction mixture on the filter paper), catalytic filter paper **7** (placed on a glass funnel containing 2.0 mmol Mn/95cm2), R.T.

**Table S10** Influence of reaction temperature over the oxidation of benzyl alcohol to benzaldehyde catalyzed by FP@Si-MnIII-Salen-TEMPO catalytic filter papera,b

|  |  |  |  |
| --- | --- | --- | --- |
| Entry | T (°C) | Conversion (%)c | Selectivity (%) |
| 1 | R.T. | 98 | 98 |
| 2 | 50 | 98 | 96 |
| 3 | Ref. | 98 | 90 |

a Reaction conditions: Benzyl alcohol (2.0 mmol), H2O:EtOH (5.0 mL, 1:2, v/v), O2 bubbling (inside the reaction mixture on the filter paper, 1 ml/min), catalytic filter paper **7** (placed on a glass funnel containing 2.0 mmol Mn/95cm2), 37 min during 3 consecutive filtration. b For applying temperature on the reaction mixture, the Erlenmeyer flask was place on a temperature controllable water bath

**Table S11** Examination of three other set ups over the efficiency of benzyl alcohol oxidation to benzaldehyde (**9a**), benzoic acid (**10a**), and direct transformation of benzyl alcohol to *N*-benzylideneaniline (**12a**)

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| Set-up | **9a** | | **10a** | | **12a** | |
| Time (min) | Conversion (%) | Time (min) | Conversion (%) | Time (min) | Conversion (%) |
| Set up 1 | 37 | 95 | 37 | 86 | 45 | 85 |
| Set up 2 | 37 | 92 | 37 | 80 | 45 | 76 |
| Set up 3a | 37 | 98 | 37 | 93 | 45 | 96 |

Reaction conditions: The preparation of **9a**, **10a**, and **12a** were performed according to the reported procedure presented in Tables 4-6, except the how set-up of the reaction (i.e. How to use the catalytic filter paper **7** in the reaction)

Set up 1: The catalytic filter paper with 5.5 cm in diameter was cut into small 1 cm-square pieces and added to the reaction mixture like a heterogeneous catalyst.

Set up 2: The catalytic filter paper with 5.5 cm in diameter was cut into 8 conical pieces the size of a 2.75 cm rim and held suspended by a clamp inside the reaction mixture.

Set up 3: The reaction was performed *via* the filtration method and O2 inlet flow (1.0 ml/min) was used as an oxidant. Number of filtration for the preparation of **9a**, **10a**, and **12a** was equal to 3.

**Table S12** Reusability evaluation of the catalytic filter paper **7** towards the oxidation of benzyl alcohol to benzaldehyde and benzoic acid in the presence of molecular O2 and NaOCl respectivelya

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
|  | Entry | Benzaldehyde | | Benzoic acid | |
| Conversion (%) | Selectivity (%) | Conversion (%) | Selectivity (%) |
| 1th cycle | 97 | 97 | 95 | 94 |
| 2th cycle | 97 | 97 | 93 | 92 |
| 3th cycle | 96 | 97 | 92 | 88 |
| 4th cycle | 96 | 97 | 92 | 86 |

a All the reusability reactions were studied for 3 consecutive filtrations for 37 min.

**Table S13** Stability study of the FP@Si-MnIII-Salen-TEMPO against acidic, basic, and oxidative reagentsa

|  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- |
| Entry | Sample | Reagent | EDX or ICP analysis (%wt of element) | | | | |
| C | N | Mn | Si | O |
| 1 | Residue  (ICP analysis) | NaOCl | - | - | 0 | 0 | - |
| 2 | HNO3 (0.1 N) | - | - | 4.56 | 6.32 | - |
| 3 | HCl (0.1 N) | - | - | 1.12 | 0.11 | - |
| 4 | NaOH (0.1 N) | - | - | 0 | 0 | - |
| 5 | H2O2 37% | - | - | 0 | 0 | - |
| 6 | FP@Si-MnIII-Salen-TEMPO  (EDX analysis) | NaOCl | 50.60 | 7.53 | 2.40 | 13.65 | 25.82 |
| 7 | HNO3 (0.1 N) | 52.76 | 3.75 | 0.26 | 13.05 | 30.18 |
| 8 | HCl (0.1 N) | 50.62 | 7.60 | 2.41 | 13.89 | 25.48 |
| 9 | NaOH (0.1 N) | 50.68 | 7.62 | 2.45 | 13.79 | 25.51 |
| 10 | H2O2 37% | 50.66 | 7.60 | 2.39 | 13.85 | 25.50 |

a For each reagent, 5 ml was used and the analyzes were taken after 10 consecutive filtrations at room temperature.



**Scheme S2** Chemoselectivity behavior of FP@Si-MnIII-Salen-TEMPO filter papertowards direct and selective oxidation of benzyl alcohol to benzoic acid in the presence of amine or hydroxylamine hydrochloride



**Scheme S3** Direct transformation of benzyl alcohol to the corresponding carboxylic acid in the presence of NaOCl catalyzed by FP@Si-MnIII-Salen-TEMPO catalytic filter paper



**Scheme S4** Hot filtration test on the FP **7**

**References**

[1] M. Kazemnejadi, S.A. Alsvi, Z. Rezazadeh, M.A. Nasseri, A. Allahresani and M. Esmaeilpour, *Green Chem*., 2019, **21**, 1718-1734.