Supporting Information

In situ growth of fluorescent silicon nanocrystals in monolithic microcapsule as a photostable, versatile platform

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Equation S1

$$(RO)_3SiR' + H_2O \rightarrow HO(RO)_2SiR' + ROH$$
$$(RO)_3SiR' + HO(RO)_2SiR' \rightarrow R'(RO)_2SiOSi(RO)_2R' + ROH$$
$$R'(RO)_2SiOH + HO(RO)_2SiR' \rightarrow R'(RO)_2SiOSi(RO)_2R' + H_2O$$

Where R' stands for aminopropyl side chain, and R represents ethyloxy group on APTES.

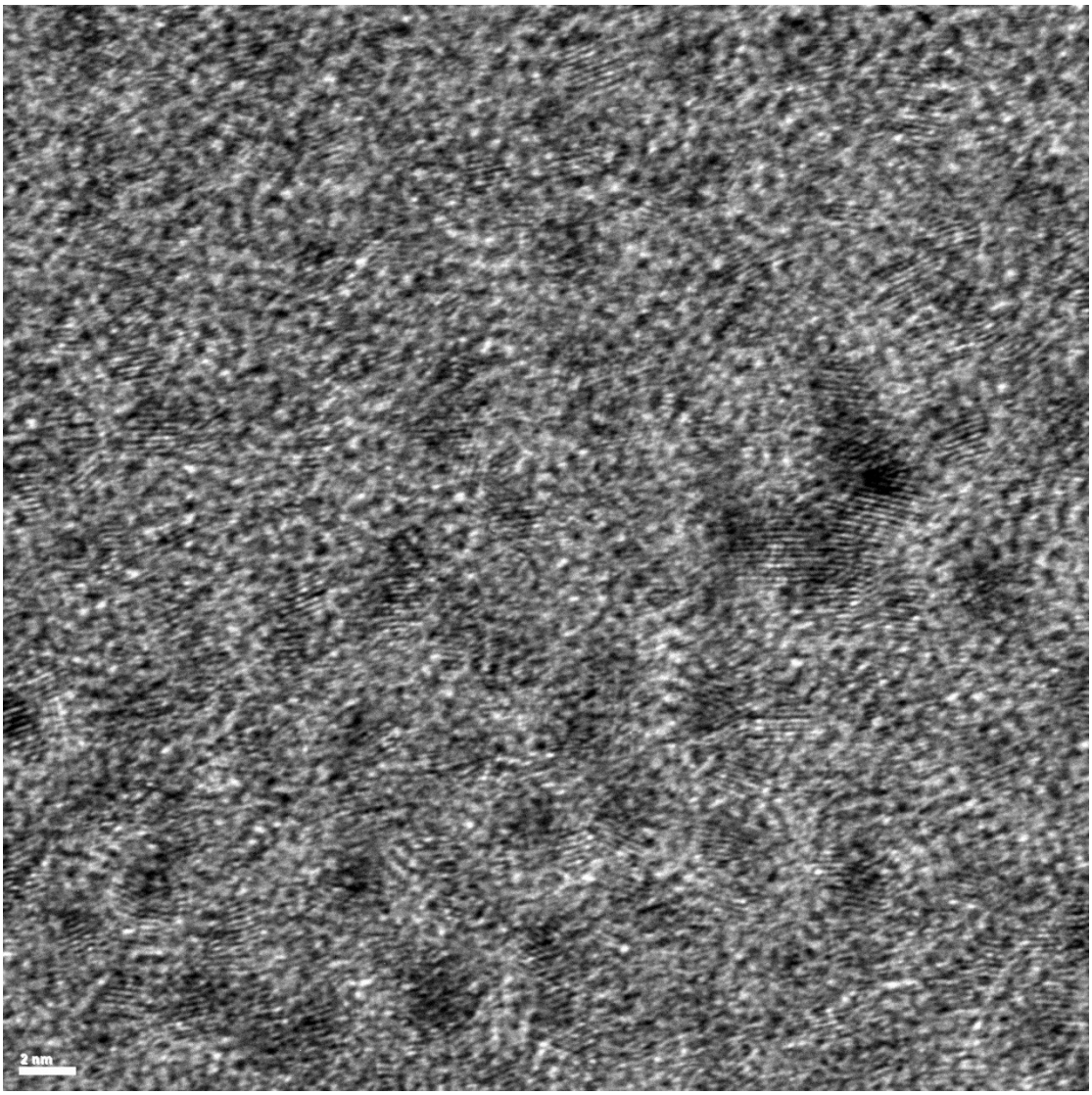

Fig. S1 TEM image of SiPM slices (70nm, cut by ultramicrotome) with well-distributed SiNC inside.

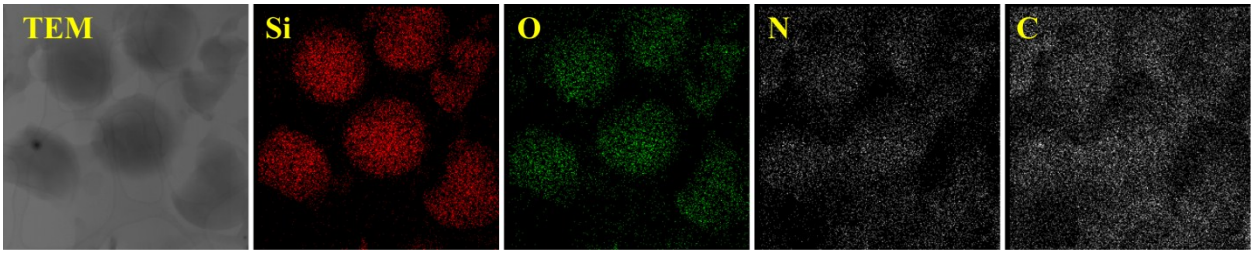

Fig. S2 EDS elemental mapping images for prepared SiPM slices (70nm, cut by ultramicrotome)

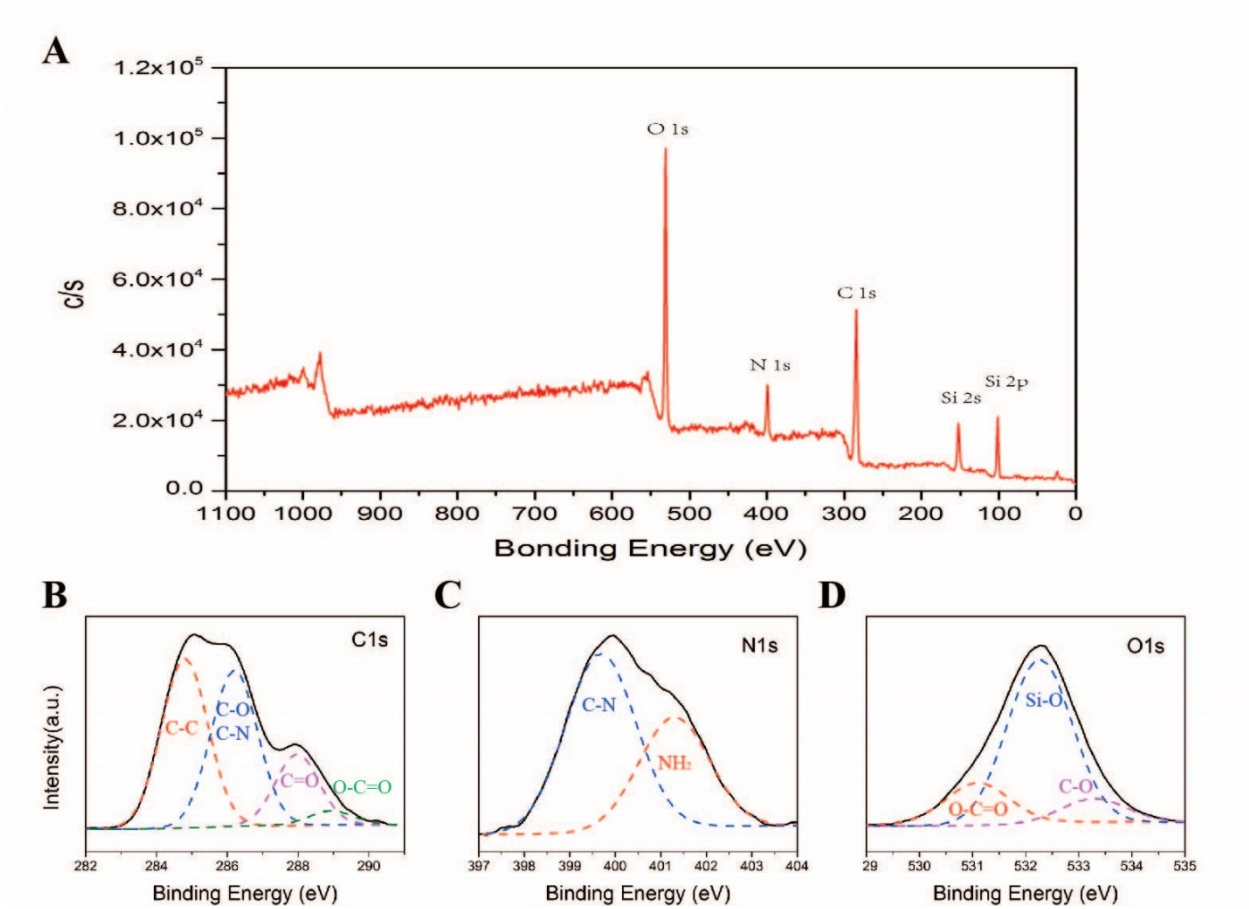

Fig. S3. XPS spectrum of SiPM (A), and structure analysis of high resolution XPS spectra: (B) C1s, (C) N1s and(D) O1s

The high solution XPS spectra indicate that various chemical bonds are contained in SiPM such as C-C, C-N, C-O, C=O and Si-O. The C=O may come from ascorbic acid in polymer matrix. The existence of Si-O, C-N and NH2 is attributed to siloxane matrices derived from APTES.

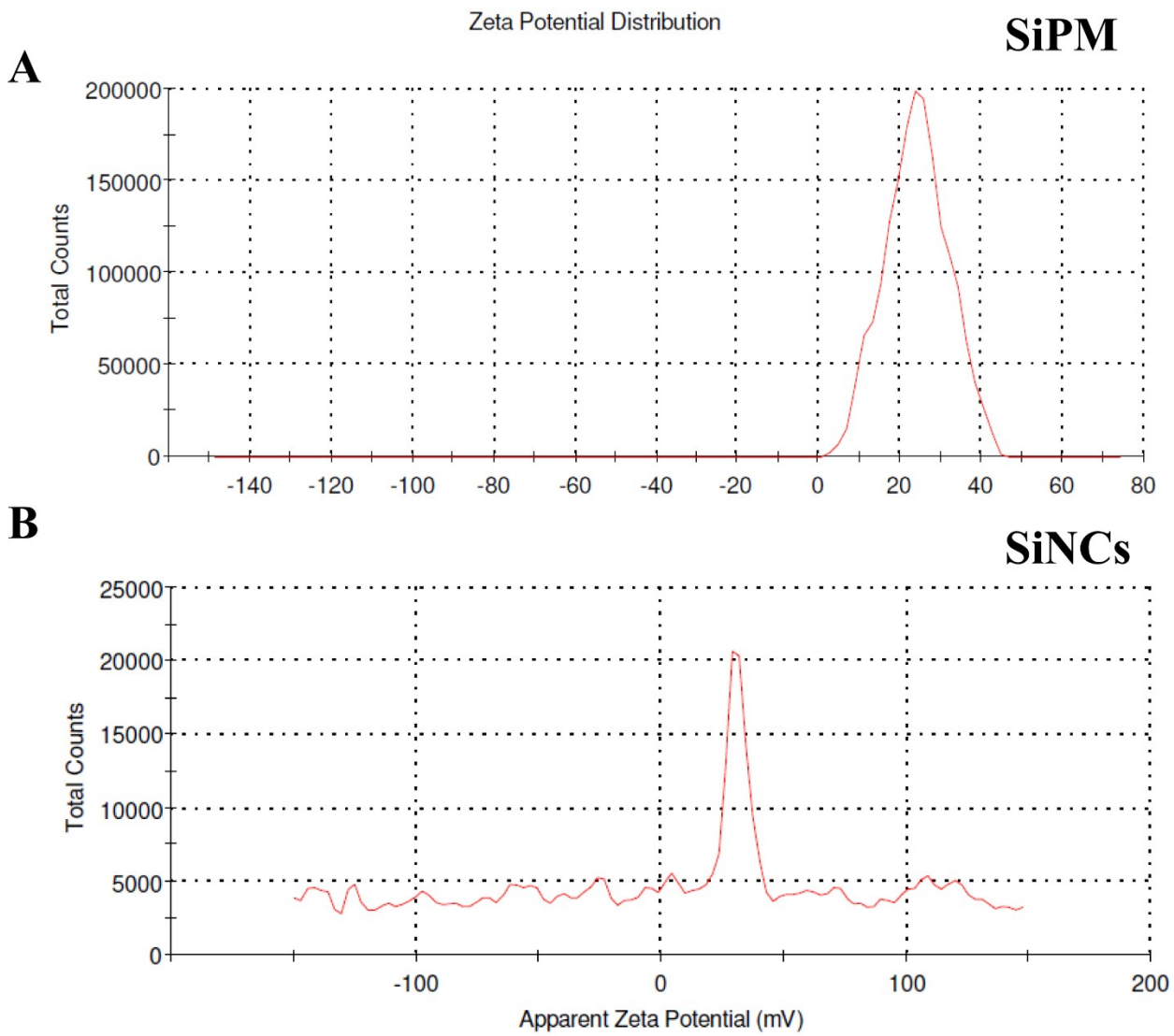

Fig. S4. Zeta potential measurement of (A)SiPM (~23mV) , and (B) hydrolytic silicon nanocrytals (SiNCs, ~29mV).

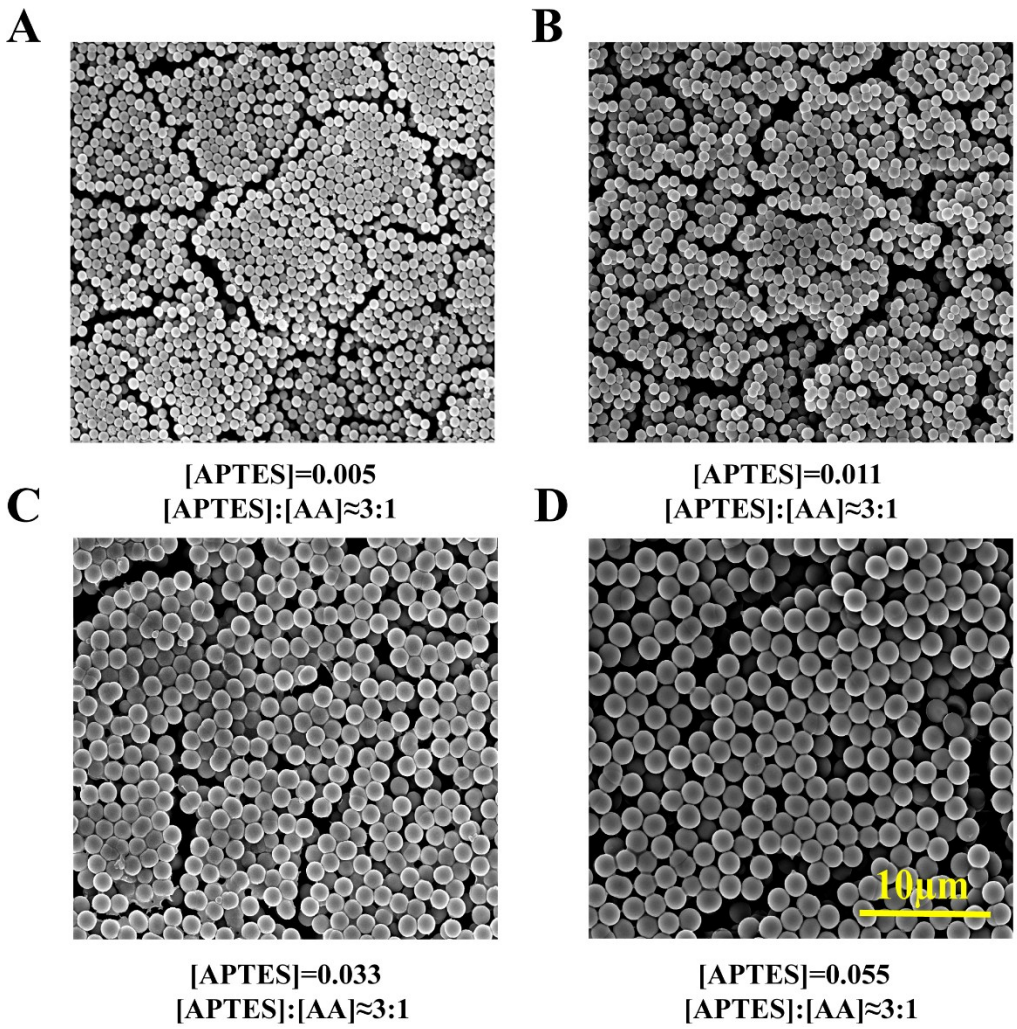

Fig. S5. SEM images of SiPM prepared at different APTES concentrations (M) in system. Scale bar is 10 μm for all images.

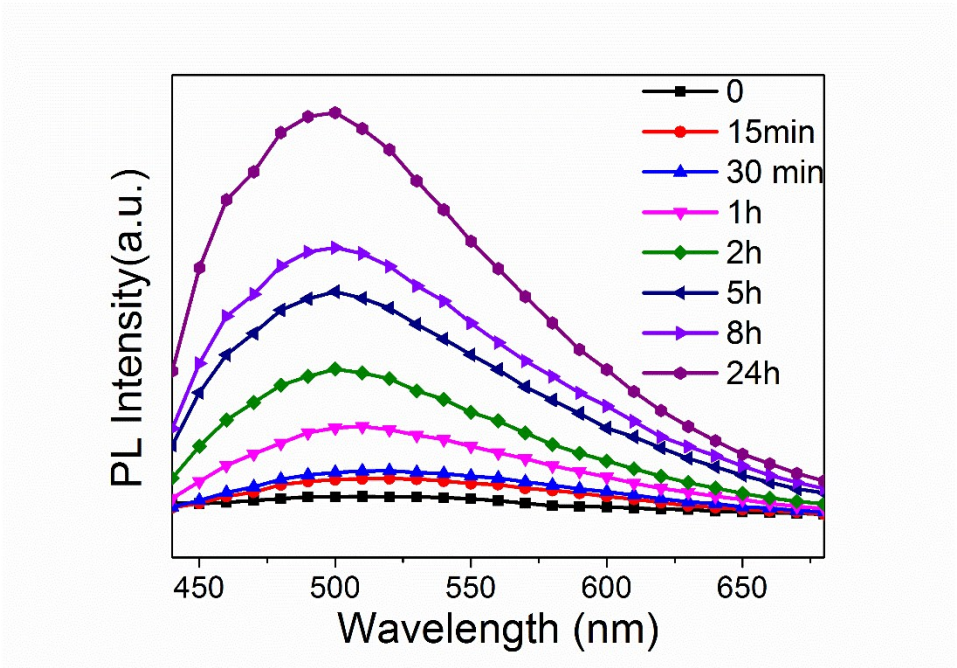

Fig. S6. The fluorescence spectra (excitation wavelength: 420nm) of SiPM with different reaction time.

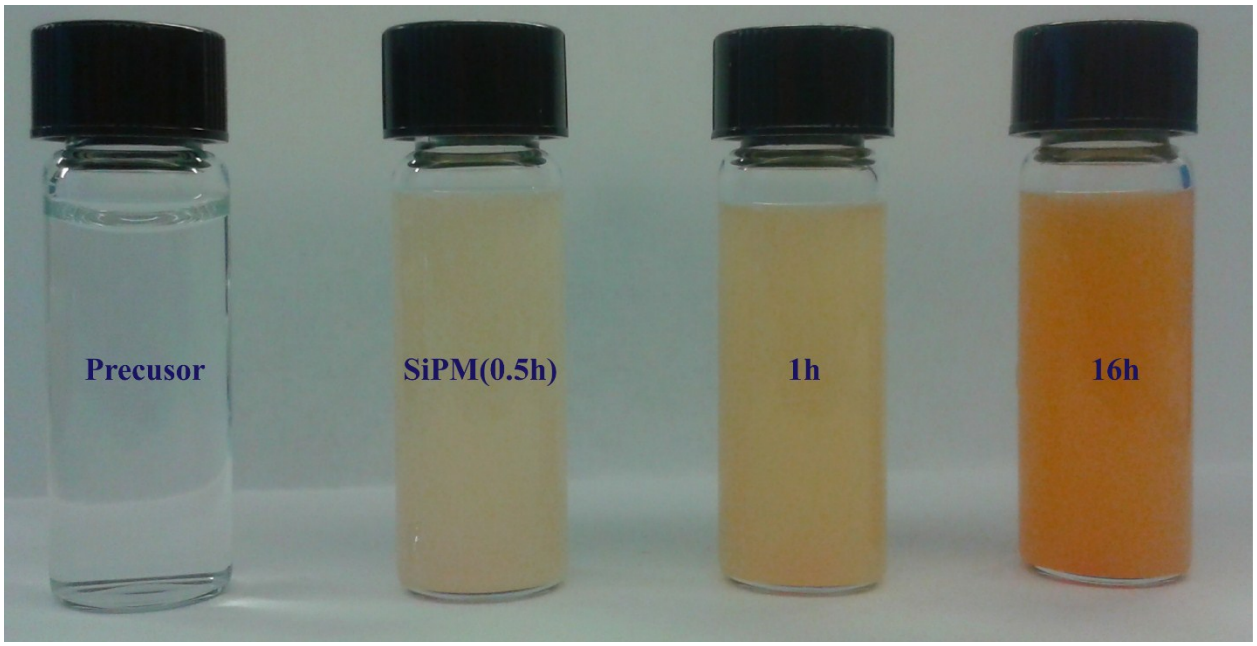

Fig. S7. Digital pictures of SiPM with different reaction times.

Table S1. Elemental composition of SiPM with different reaction time

	C1s (%)	N1s (%)	O1s (%)	Si2p (%)
SiPM (30min)	48.57	10.47	29.70	11.26
SiPM (16h)	42.85	7.57	33.40	16.19

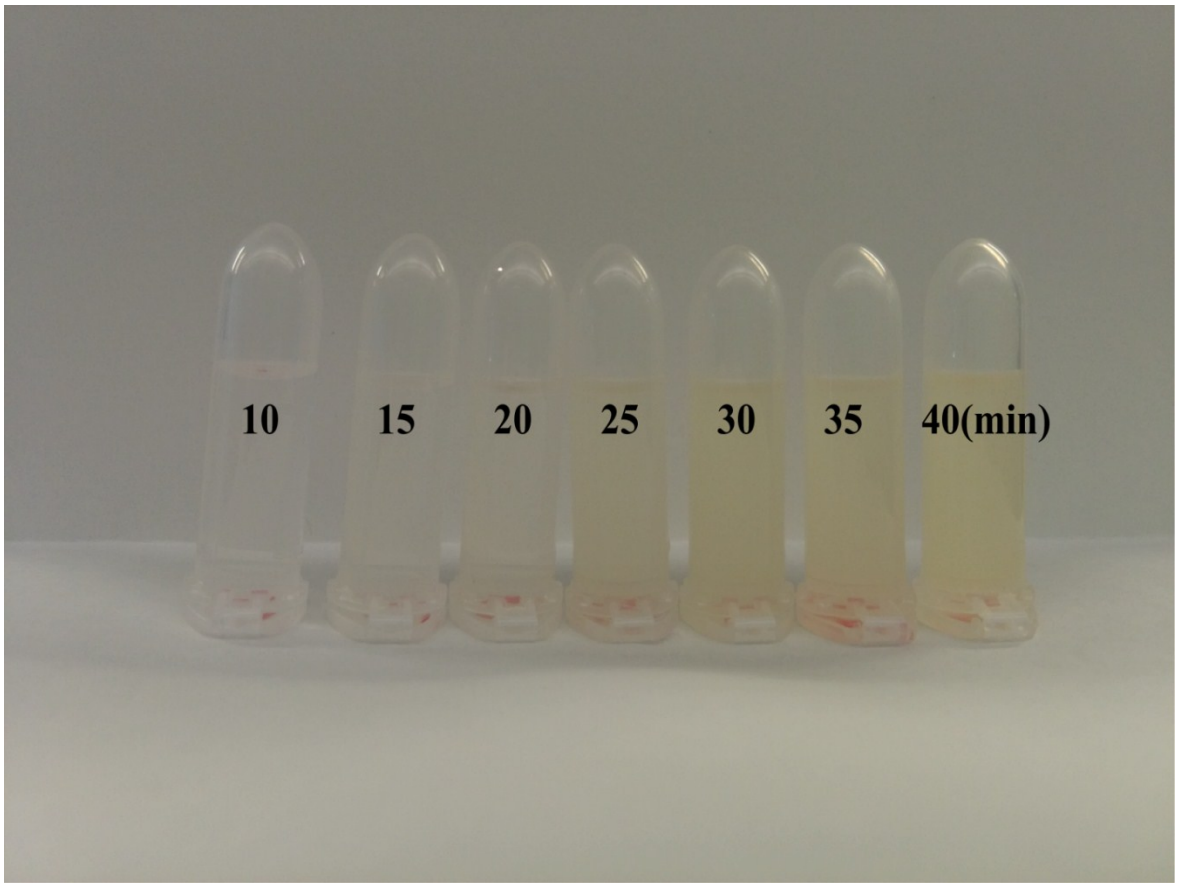

Fig. S8. Pictures of SiPM in tubes with increasing reaction time.

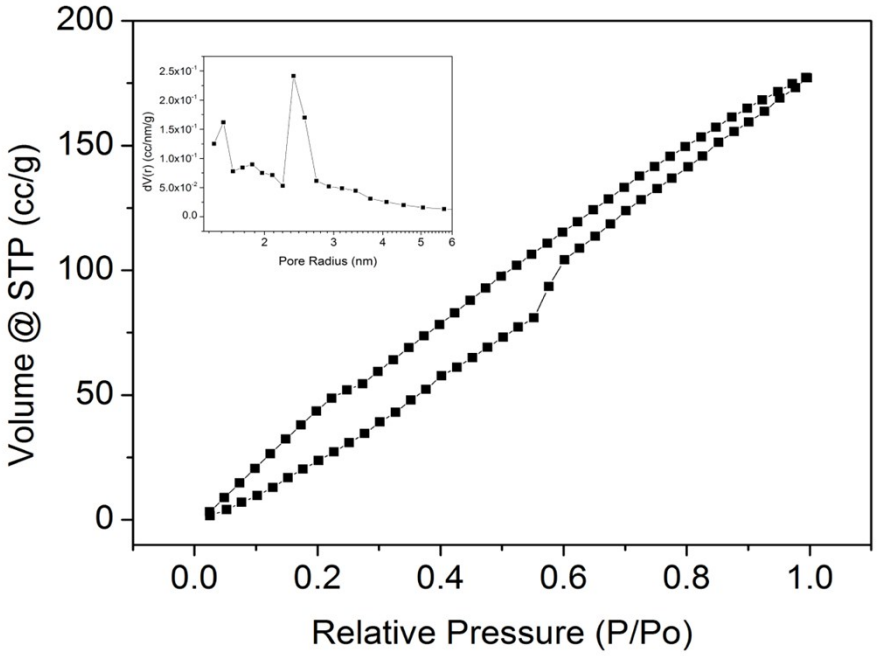

Fig. S9. N2 adsorption/desorption isotherms of LD-SiPM (30min reaction time), inset displays Barrett-Joyner-Halenda (BJH) pore-size distribution curve obtained from the adsorption branch.

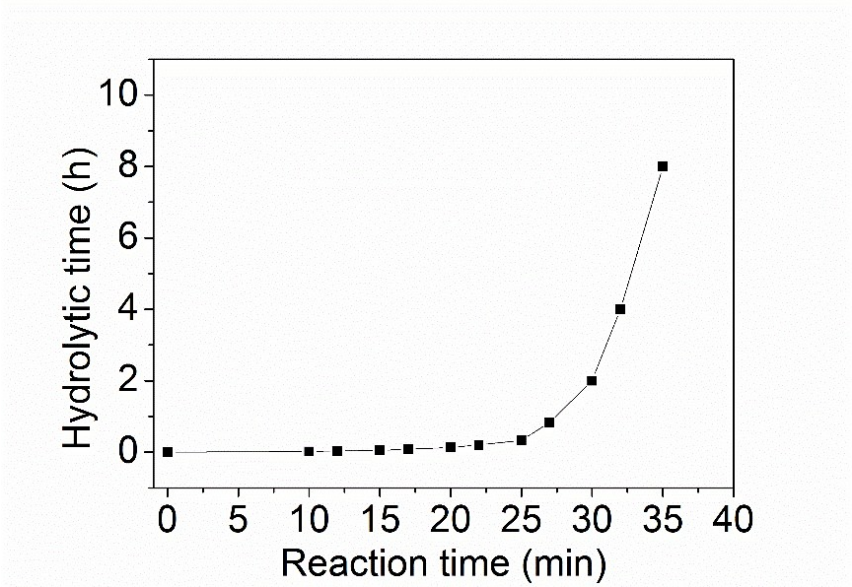

Fig. S10. Correlation of hydrolytic time of SiPM dispensed solutions (in PBS 7.4) with reaction time

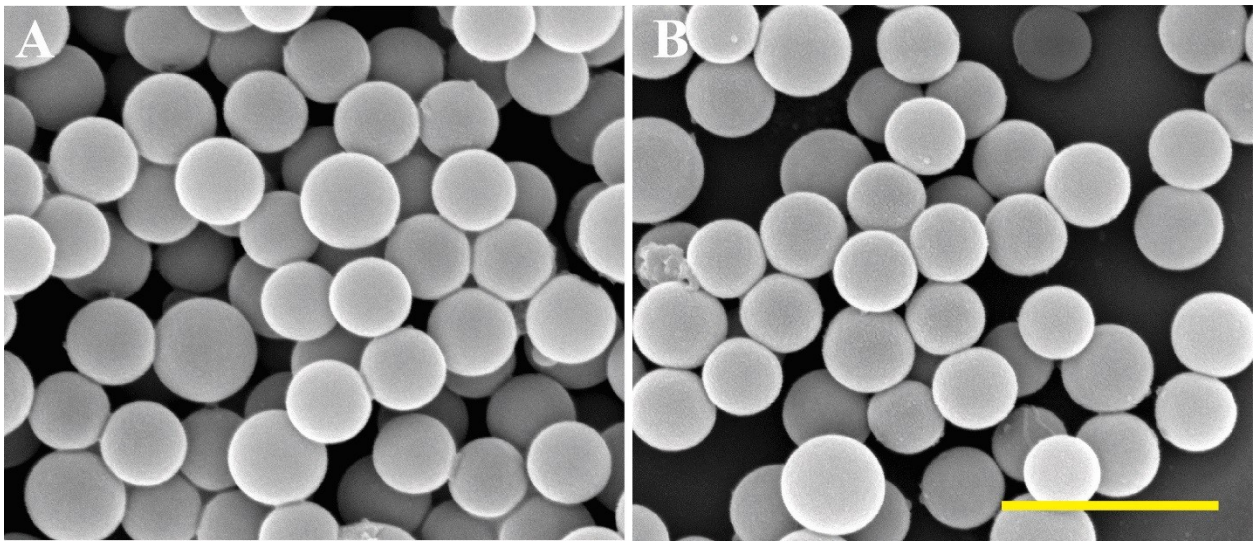

Fig. S11. SEM images of HD-SiPM after immersing in: (A) PBS (pH=7.4), and (B) water for three months.

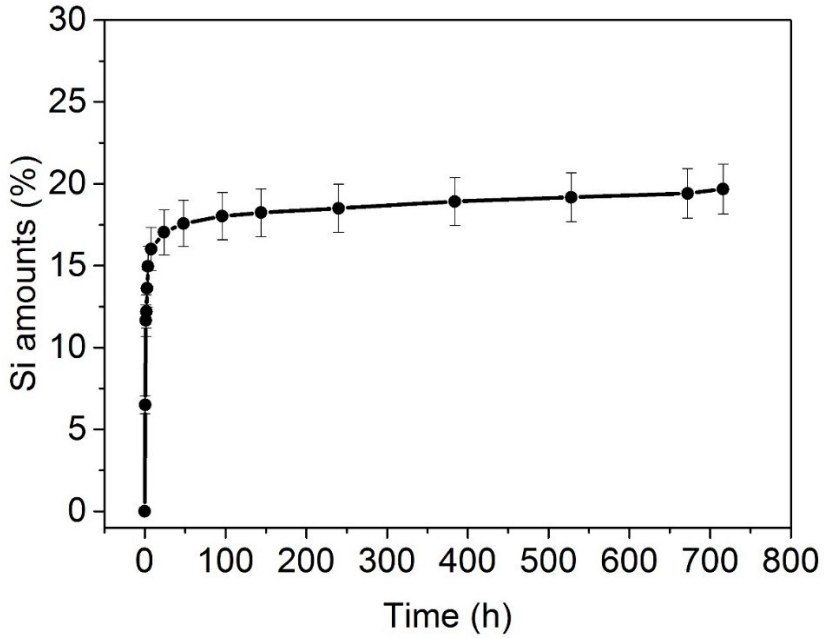

Fig. S12. Releasing curve of silicon elements (measured by ICP/OES) from SiPM (reaction time 1h) versus time in PBS (pH=7.4) at 37 °C.

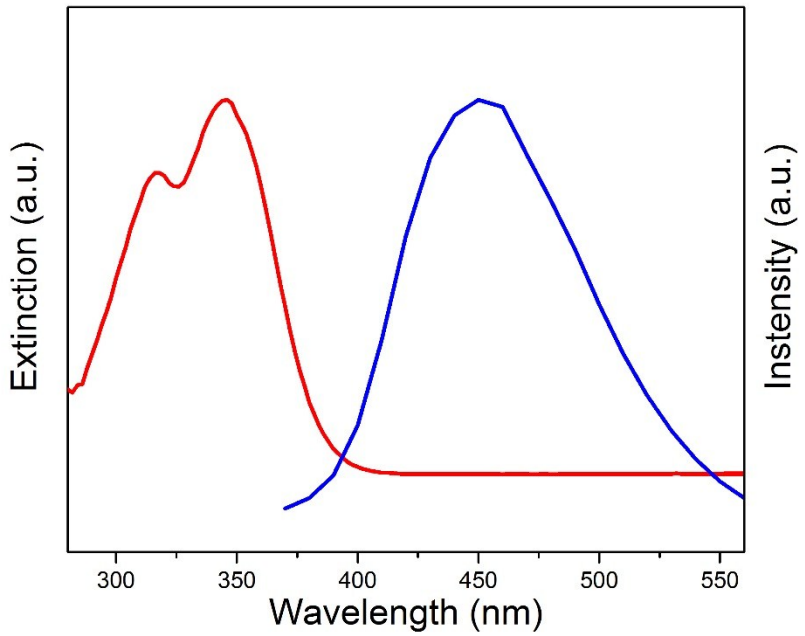

Fig. S13. UV-vis absorbance and photoluminescence curve of quinine sulfate.

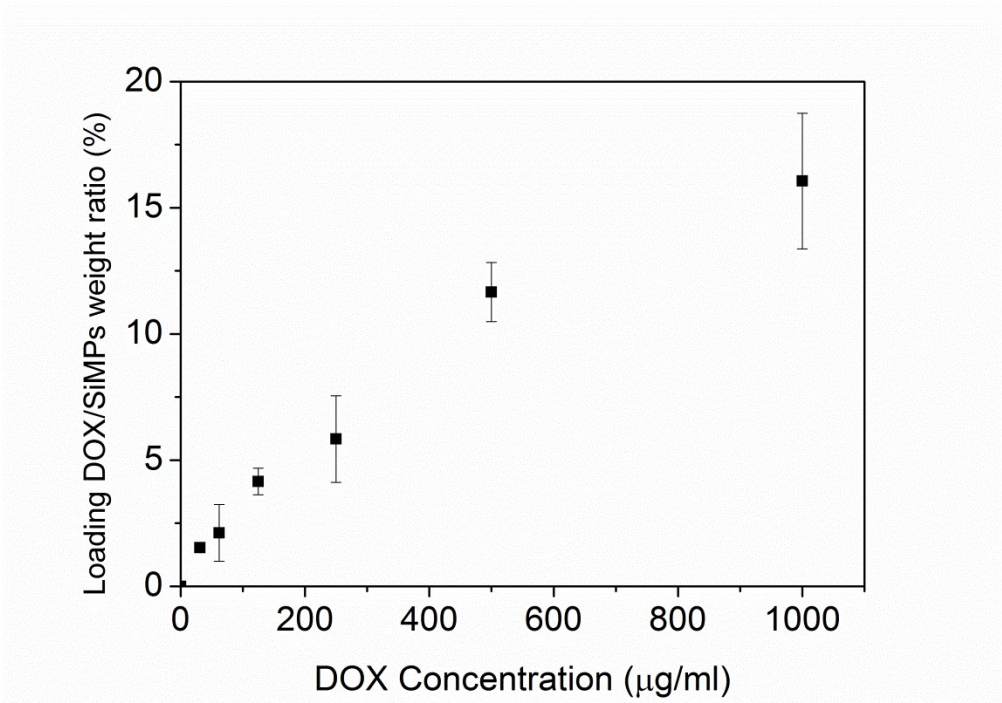

Fig. S14 Quantification of DOX loading in SiMP with various concentrations.

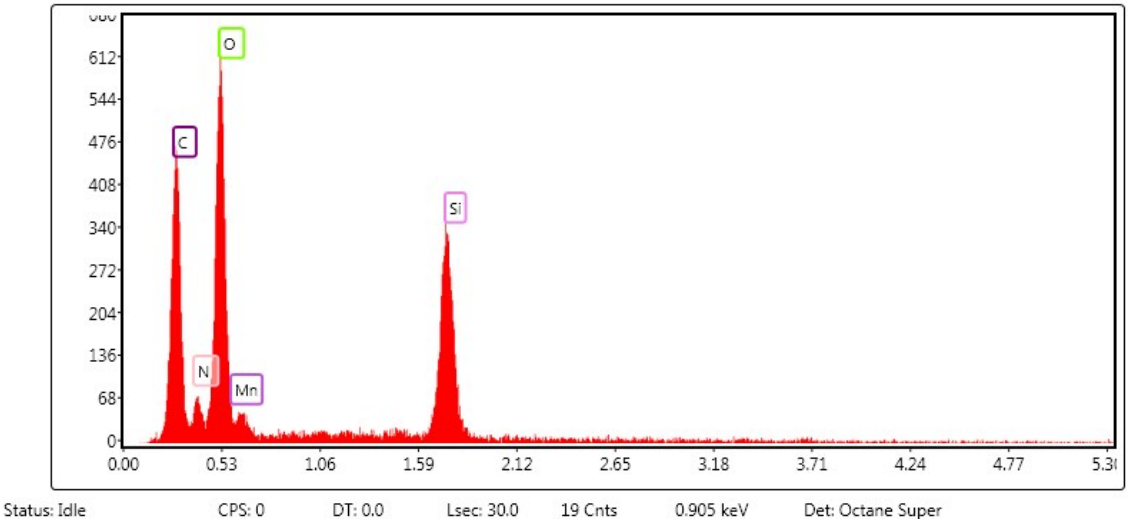

Fig. S15. EDS spectrum of SiPM/MnO2 compisite

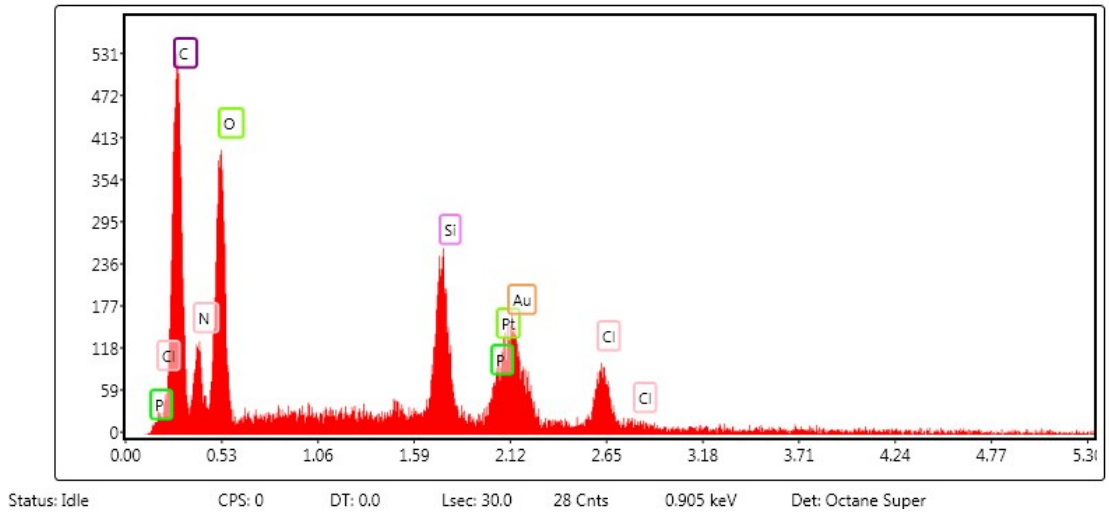

Fig. S16. EDS spectrum of SiPM/Au compisite

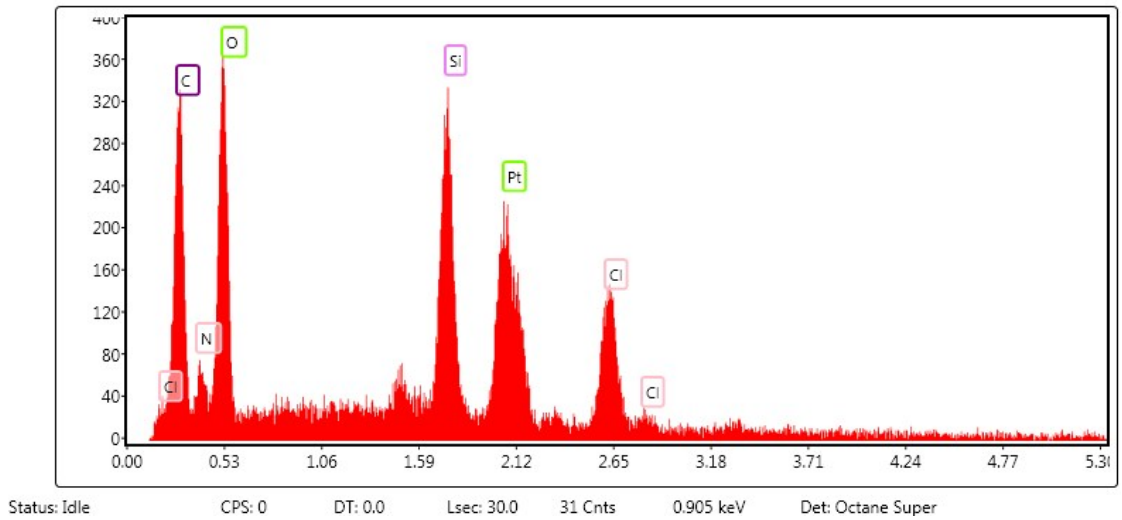

Fig. S17. EDS spectrum of SiPM/Pt compisite

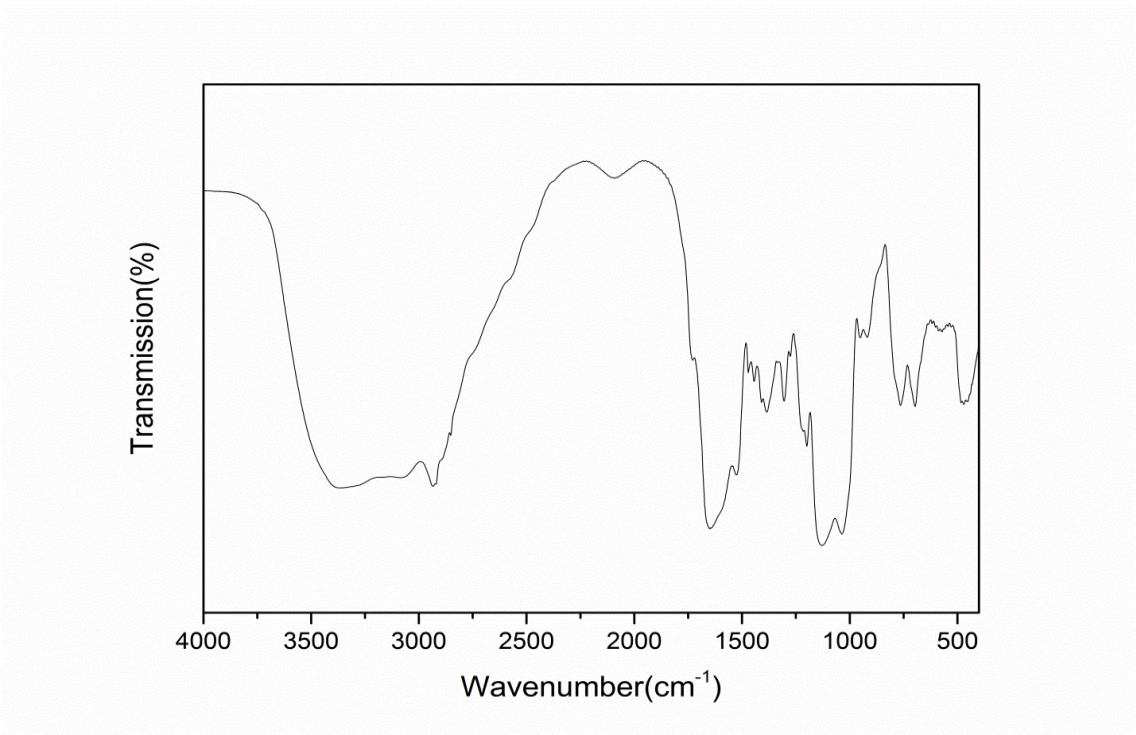

Fig. S18 FTIR spectrum of SiPM

The FTIR exhibits distinct peaks ranging from 1000 to 3500cm-1. The peaks around 1000cm^-1 and 1200cm^-1 are characteristics of stretching vibrations of siloxane (Si-O-Si) groups. The absorbance peak at 2938cm^-1 is due to C-H stretching. The broad peak between 3000cm^-1 to 3600cm^-1 stands for O-H stretching. The signals at 3400cm^-1 and around 1637cm^-1 are attributed to stretching and bending of N-H bond. Strong conjugates effects and hydrogen bonding could lower the frequency of C=O stretching vibration and enhance its absorbance. The sharp peak at 1637cm^-1 would be the overlapping signals of N-H, C=C and C=O stretching vibrations.

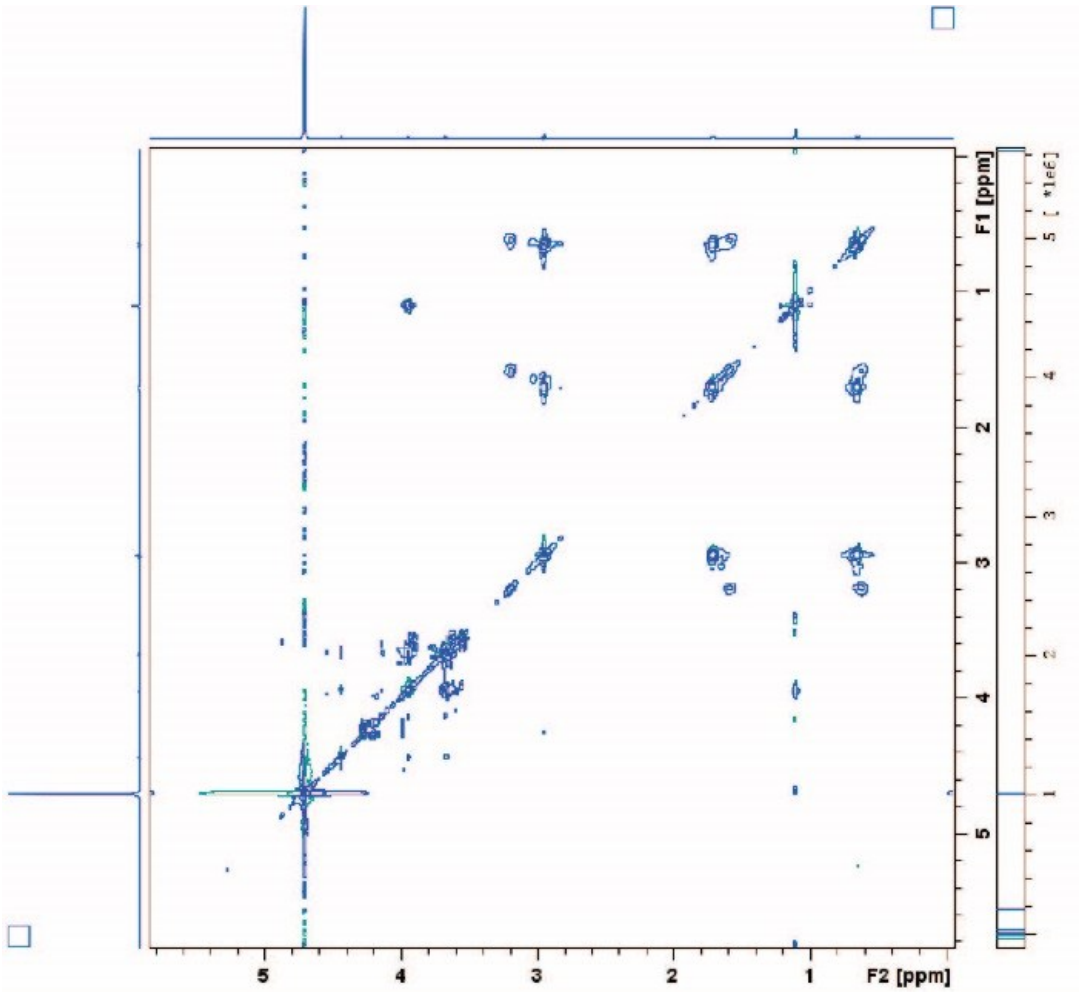

Fig. S19 2D TOCSY NMR spectrum of hydrolytic SiPM in D2O.

2D-TOCSY spectrum was measured in ordering to know the chemical structure of polymer matrices in SiPM. There is no obvious coupling between APTES and ascorbic acid. We supposed that the non-covalent bonding, electrostatic adherence or hydrogen bond might be the main boding method in the polymer matrix in SiPM.

(1) A. Shiohara, S. Hanada, S. Prabakar, K. Fujioka, T. H. Lim, K. Yamamoto, P. T. Northcote and R. D. Tilley, Chemical Reactions on Surface Molecules Attached to Silicon Quantum Dots. *J Am Chem Soc* **2010,** *132* (1), 248.
(2) S. E. Lehman, Y. Tataurova, P. S. Mueller, S. V. S. Mariappan and S. C. Larsen, Ligand Characterization of Covalently Functionalized Mesoporous Silica Nanoparticles: An NMR Toolbox Approach. *J Phys Chem C* **2014,** *118* (51), 29943.
(3) R. S. Reid, The Proton Nmr-Spectrum of Ascorbic-Acid - a Relevant Example of Deceptively Simple 2nd-Order Behavior. *J Chem Educ* **1989,** *66* (4), 344.