Electronic Supplementary Material (ESI) for Dalton Transactions.

Detail explanation on procedure of deconvolution of Raman spectra

'Deconvolution' is the method of involving the process of decomposing peaks that overlap with each other. It is required to extract the information about the hidden peak/peaks. The no. of experimentally observed Raman modes are significantly lower than that of predicted symmetry since a large number of vibration modes cannot be separated within the resolution of the Raman spectrometer. The several weak intense modes are usually overlapped with the neighboring prominent modes and cause broadening. To extract out the contribution of each Raman mode from the broadened Raman peak, the method of deconvolution was employed. The process of deconvolution of the Raman peak is authentic and earlier been used by several researchers [A A Porporati et al J. Raman Spectrosc. 41, 84–87(2010), S. Chaturvedi et al J. Mater. Chem. C 4, 780-792(2016), M. Nadeem et al J. Appl. Phys. 124, 164105 (2018), S. Sayyed et al RSC Adv. 5, 50644-50654(2015)].

The deconvolution method was performed using OriginPro 8.5 software. The Raman spectrum was deconvoluted using the Gaussian fitting function. The stepwise detailed procedure of deconvolution can be elucidated as ;

- **Baseline treatment**

  The raw data comes with non-constant baseline. Therefore, the baseline to the Raman peaks was detected and treated.

- **Deconvolution of spectra**

  The entire broadened peak (where the possibility of overlapping of no. of peaks) was selected. Manually, we have added/specified the number and position of the 'hidden' peaks (the number and position were determined from literature). Then, we have defined the peak function (i.e. Gaussian) and the no. of iterations were performed until the overall fit is converged. The quality of deconvolution can be decided with the value of reduced χ^2 and typically, it should be closer to 1. Reduced χ^2 can be defined as;

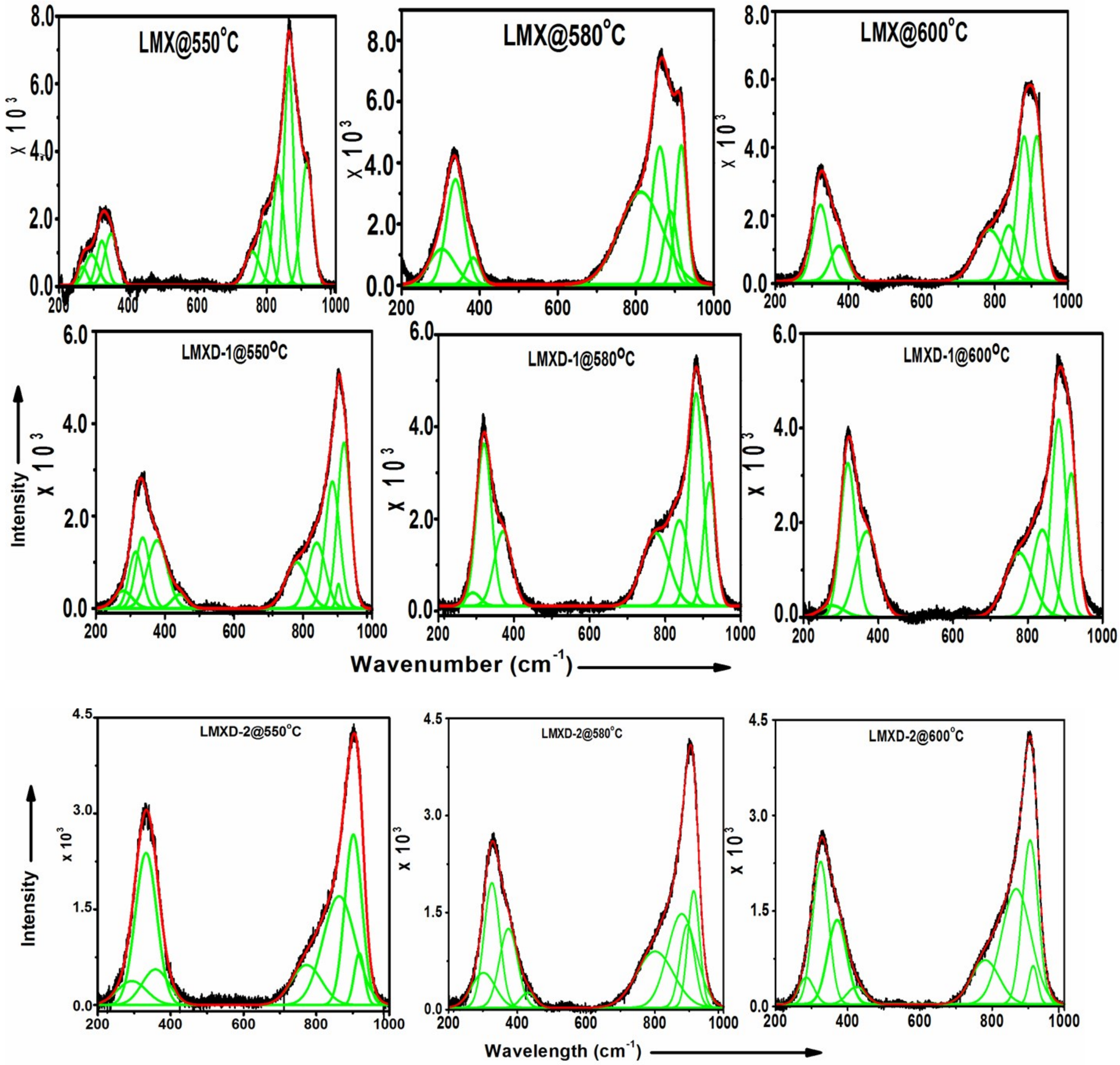
$$Reduced\chi^2 = \frac{1}{(n - p)}\sum_{i=1}^{n}\frac{(y_i - f_i)^2}{\sigma_i^2} \qquad \text{.....................(1)}$$

  where *n* is the number of data, *p* the degree of freedom, y_i the i^th^ *y* data, and σ_i the ith error. When the number of data is large enough, *n - p* approaches *n*,

and reduced χ^2 is determined by the difference between source data and fitted data and the weight.

We have repeatedly performed deconvolution by changing the number and position of hidden/overlapped peaks. We have come up with the reduced χ^2 value in the standard range for the specific number of deconvoluted peaks and inferred that this the only possible deconvolution.

2. Fig S1-Deconvoluted data of Raman spectra at 550, 580 and 700°C of LMX, LMXD-1, LMXD-2, LMXD-3



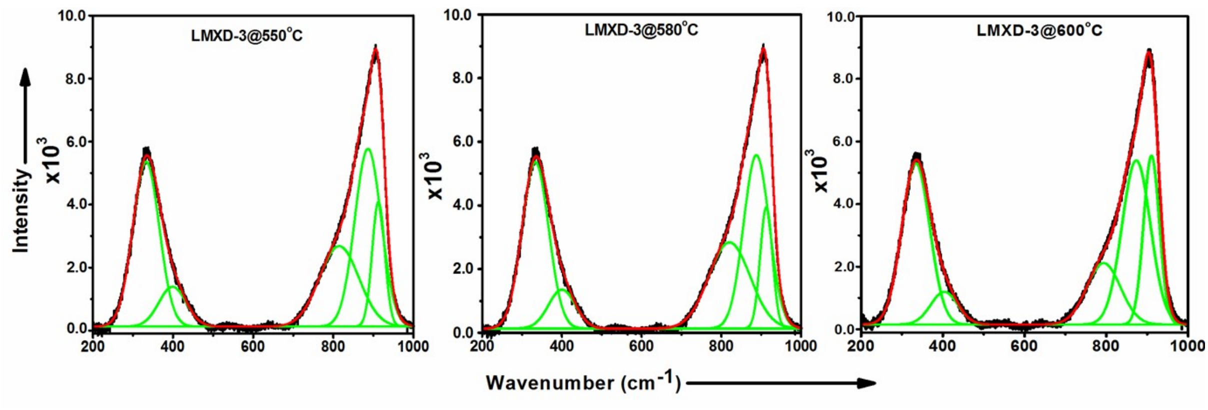


Fig S2- Magnified view of High temperature Raman spectra of LMX.