Experimental Theory and Design

Introduction

There are several steady-state and transient techniques available for the measurement of thermal properties. Although not as accurate as steady-state methods such as the Guarded Hot Plate apparatus, transient techniques offer important advantages over steady-state techniques. These include simultaneous measurement of thermal conductivity and thermal diffusivity, significantly shorter duration of experiments and capability of studying moisture effects on thermal properties. Moreover, transient measurement setups are generally simpler and could be built at a lower cost compared to the steady-state counterparts.

The transient measurement methods [1]are distinguished by the type (wire, strip or plane) and thermal profile (pulse or step) of heat source and the point of temperature measurement (at source or away from the source). One of the first techniques was implemented by Gustafsson [2] in 1991 using a Transient Plane Source element (now known as the "Gustafsson Probe") which works as both the heat source and the sensor. Transient method involving step-wise heating was discussed by Kubicar and Bohac [3]. Lei et al. [4] used the transient plane source method with stepwise heating to measure thermal properties of fabrics which are fibrous in nature. Recently, Malinari [5] carried out a detailed analysis of Step-wise transient method with disk shaped plane heat source.

Measurement Theory

In the current work, Transient Plane Source (TPS) with a step heat flux is used for the measurement of thermal conductivity and thermal diffusivity. The temperature profile is captured by a sensor positioned at a fixed distance away from the heat source.

Semi-infinite Model

The experimental setup is designed to replicate the 1-D semi-infinite transient model illustrated in Figure 1 during the duration of the experiment. The analytical solution for the model is given by,

$$T(x,t) = \frac{2q}{k}\left[\sqrt{\frac{\alpha t}{\pi}}\exp\frac{-x^2}{4\alpha t} - \frac{x}{2}\operatorname{erfc}\left(\frac{x}{2\sqrt{\alpha t}}\right)\right] \tag{1}$$

where q is the heat flux and x is the distance between the sensor and the heat source. The sample can be approximated as a semi-infinite solid to a certain degree of accuracy if the following criterion is satisfied:

$$Fourier Number \;\; Fo = \frac{\alpha t}{(L/2)^2} \leq 0.2 \tag{2}$$

where, t is the measurement time and L is the thickness of the sample. An approximate maximum value of thermal diffusivity, α_{max} (obtained from literature or other sources) for the material of the sample or similar material shall be used in the above condition. By setting a practical value of sample thickness, the maximum duration of the experiment is obtained as follows.

$$t_{max} = \frac{0.2(L/2)^2}{\alpha_{max}} \tag{3}$$

The side length W of the square shaped sample is fixed to be atleast five times the distance between the heat source and the temperature sensor as suggested by Tye et al. [].

The temperature profile obtained from the experiment is curve-fit to the analytical solution to get k and α using non-linear parameter estimation procedure.

Sensitivity Coefficient Analysis

The selection of the time window to be used for the curve fitting procedure depends on the sensitivity of the parameters to the change in temperature as well as the linear dependence of the parameters to each other. Sensitivity coefficient analysis is to be carried out for this purpose.

Sensitivity Coefficient is defined as

$$\beta_p(t) = p\frac{\partial T}{\partial p} \tag{4}$$

where p is an unknown parameter. If the sensitivity coefficients are low or linearly dependent on each other, the curve fit may not work properly. Here, the unknown parameters are k and α for which the corresponding sensitivity coefficient expressions are given by,

$$\beta_k = \frac{-2q}{k}\left[\sqrt{\frac{\alpha t}{\pi}}\exp\frac{-x^2}{4\alpha t} - \frac{x}{2}\,\mathrm{erfc}\,(\frac{x}{2\sqrt{\alpha t}})\right] \tag{5}$$

$$\beta_\alpha = \frac{q}{k}\sqrt{\frac{\alpha t}{\pi}}\exp\frac{-x^2}{4\alpha t} \tag{6}$$

The plot of β_k and β_α against time or Fo indicates the initial period of time during which the properties are not sensitive to the change in temperature because of which their values cannot be estimated accurately. Since the less diffusive materials (low α) tend to take longer to respond to the applied heat flux, we shall consider an approximate value of α_{min} of the sample under study. To apply the sensitivity analysis for different materials, we make use of the normalized forms of the sensitivity coefficients defined as,

$$\beta_p^{'} = \left|\frac{\beta_p - \beta_{p,0}}{\beta_{p,max} - \beta_{p,0}}\right| \tag{7}$$

where $\beta_{p,0}$ is the sensitivity coefficient value at the start of the measurement (at time $t = 0$) and and $\beta_{p,max}$ is the same at the end of the measurement (at time $t = t_m$)

A set of parameters are said to be linearly dependent if they satisfy the condition,

For the unknown parameters k and α, we can write

$$C_1\frac{\partial T}{\partial k} + C_2\frac{\partial T}{\partial \alpha} = 0$$

By replacing the partial derivate terms with their non-dimensional equivalents,

$$C_1\beta_k^{'} + C_2\beta_\alpha^{'} = 0$$

$$\gamma^{'}(say) = \frac{\beta_k^{'}}{\beta_\alpha^{'}} = C$$

C_1, C_2 and C are all constants. Visual inspection of the plot of $\gamma^{'}$ against the measurement time gives us some idea about the nature of the linear dependence between the two parameters.

Standard and Difference Analysis

The standard analysis is carried out by performing the curve fitting by considering the temperature data for the time interval $[t, t_{max}]$ where t is increased from 0 to $t_{max} - 1$. In difference analysis, we consider the time window $[t - t_s, t]$ where t_s is the chosen time window interval and t is again increased from t_s to t_{max}. The corresponding data is used in the curve fit procedure to obtain k and α. By selecting different values of t_s, we can arrive at the desired time window in which the influence of the linear dependence of the parameters is nullified to considerable extent.

Before the start of transient experiments, the standard and difference analyses are carried out using the temperature profile obtained from the theoretical solution (eq. 1) for each sample from which the time window is chosen. However in the actual experiments, random error in the form of noise is inevitably introduced in to the temperature data, the magnitude of which cannot be quantised accurately. Therefore, in addition to performing standard and difference analyses on the ideal temperature profile, we also consider theoretical temperature data in which random error of certain degree is introduced manually. The time window thus obtained from the error-ridden case will be greater than that obtained for the ideal case and more appropriate when planning the experiments.

Uncertainty Analysis

For calculating the standard deviation of the estimated parameters, the uncertainty analysis method described in Beck and Arnold [7] and implemented in [5] is followed here.

For the set of parameters p_j, the standard deviation of the least-square estimate is obtained as shown below:

$$\sigma^2(p_j) = \{(X^T.X)^{-1}\}_{jj}\sigma^2 \tag{8}$$

where σ is the standard deviation of the measurement system and X is the sensitivity matrix defined as,

$$\{X\}_{jj} = \frac{\partial T(t_i, \bar{p})}{\partial p_j} \tag{9}$$

Experimental Setup

The heating element consisting of a Nickel foil (25 micron thickness), having a square shape and spiral structure with thin layers of kapton on either sides acts as

the plane heat source. It is heated by a DC power source supplying constant current. Two identical samples are placed sandwiching the heat source and held under force through a pressing mechanism to ensure good contact between the sample and heating element. Heat conducting paste is also applied on the element to minimize contact resistance existing because of the presence of air gaps.

Temperature is measured using bare wire K-type thermocouples with bead diameter of about 0.5 mm positioned by appropriate methods. For the case of solid samples, a hole is drilled in the radial direction in such a way that the thermocouple is held tightly in the desired position. In the case of fibrous materials, the positioning is more difficult and hence a support made of a similar material to the sample - a thin cylindrical wooden stick with a groove to hold the thermocouple wire - is used to position the thermocouple. Once again, heat conducting paste is used to reduce the contact resistance existing between the tip of the sensor and the contact area in the sample. For all the samples considered for study here, the thermocouples are positioned 1 cm away from the source on both sides.

The entire setup is placed inside a climate chamber in order to study the effects of temperature and relative humidity on the thermal properties of the sample. Two HTU21D temperature-humidity sensors interfaced with Arduino controller are used to monitor the chamber humidity and temperature. Additionally, two thermocouples also monitor the chamber temperature. The temperature data is logged in a desktop computer through the Agilent data acquisition system.

Calibration of Equipment and Instrumentation

Calibration of the thermocouples and the relative humidity sensors were carried out before the start of the experiment.

Results and Discussion

Polymethyl Methacrylate (PMMA) and Extruded Polystyrene (XPS) are the reference samples considered for validating the experimental method while the material under study is rice paddy straw. The sample size is determined from the semi-infinite condition mentioned above. The time window (t_0 to t_m) for curve-fitting is chosen by performing the analyses discussed above for each sample. The results of the analyses are presented in Figure 2 and 3. The final experimental design values are listed in Table 1.

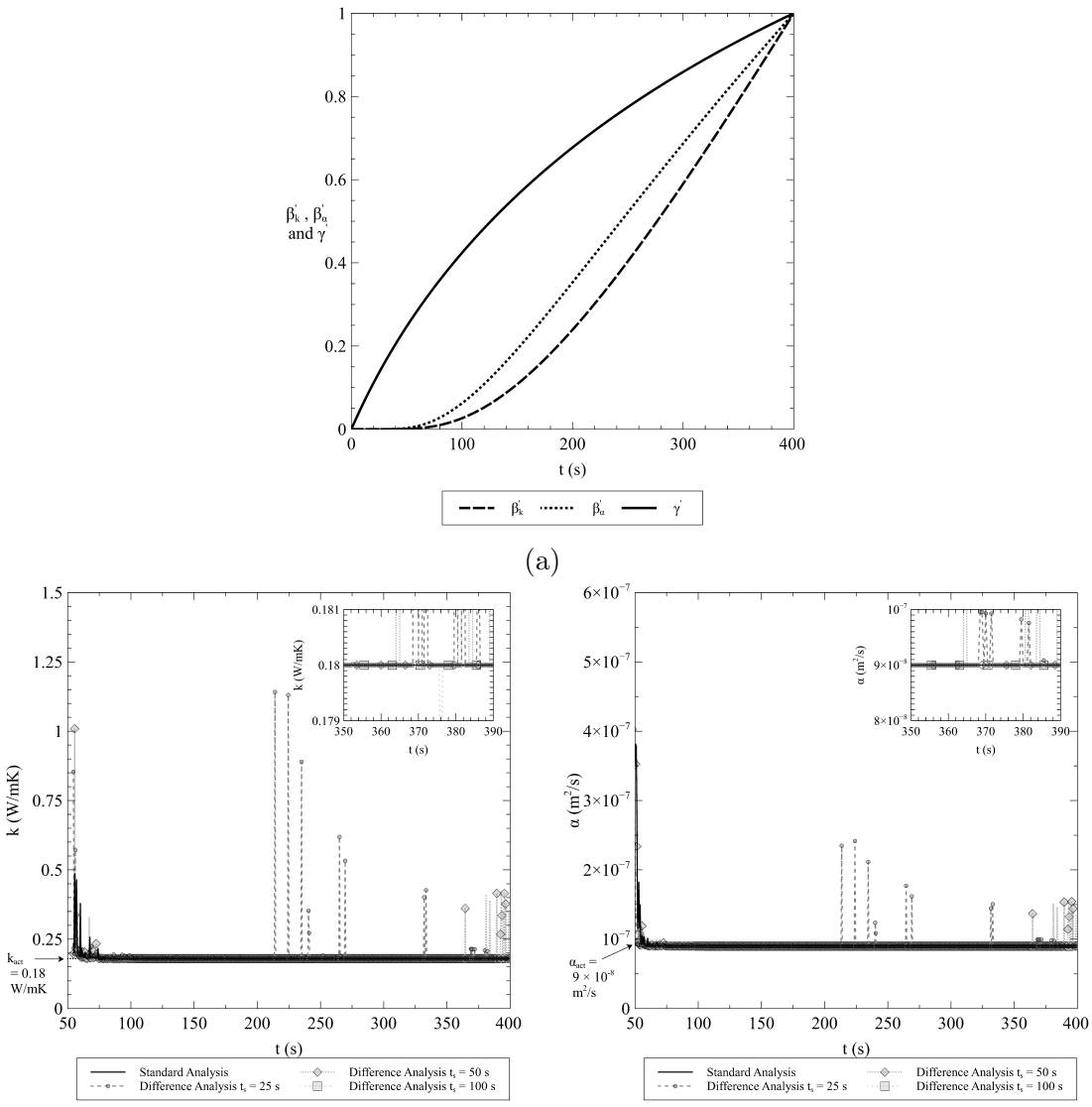

Figure 1: Measurement analysis for PMMA: (a) Sensitivity coefficients and their ratio (b) Standard analysis and difference analysis estimating thermal conductivity for three different time windows (c) Standard analysis and difference analysis estimating thermal diffusivity for three different time windows

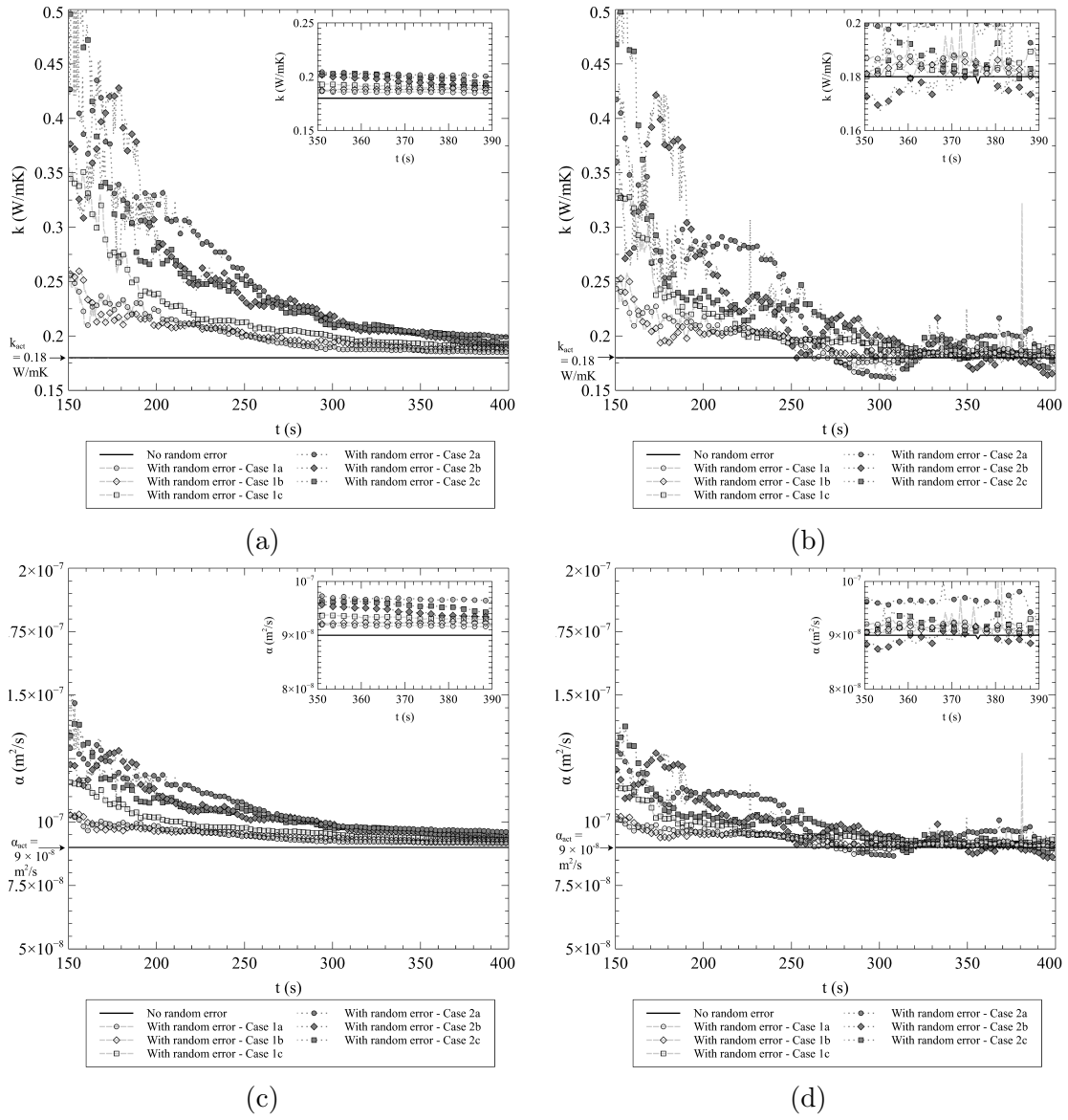

Figure 2: Measurement analysis for PMMA: (a) Standard analysis with and without random error compared for estimating thermal conductivity (b) Difference analysis with and without error compared considering time window of 100 s for estimating thermal conductivity (c) Standard analysis with and without random error compared for estimating thermal diffusivity(d) Difference analysis with and without error compared considering time window of 100 s for estimating thermal diffusivity

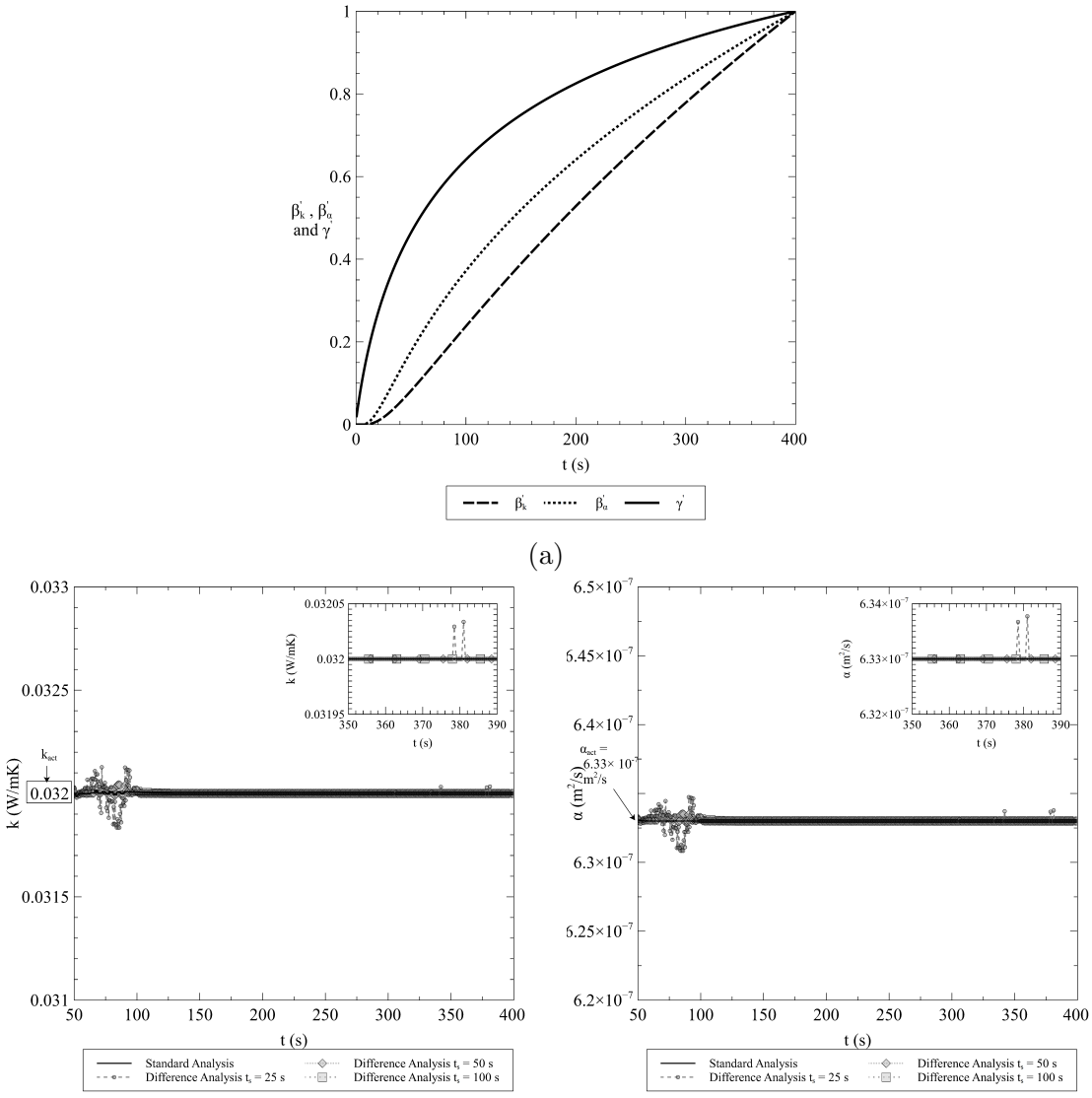

Figure 3: Measurement analysis for XPS: (a) Sensitivity coefficients and their ratio (b) Standard analysis and difference analysis estimating thermal conductivity for three different time windows (c) Standard analysis and difference analysis estimating thermal diffusivity for three different time windows

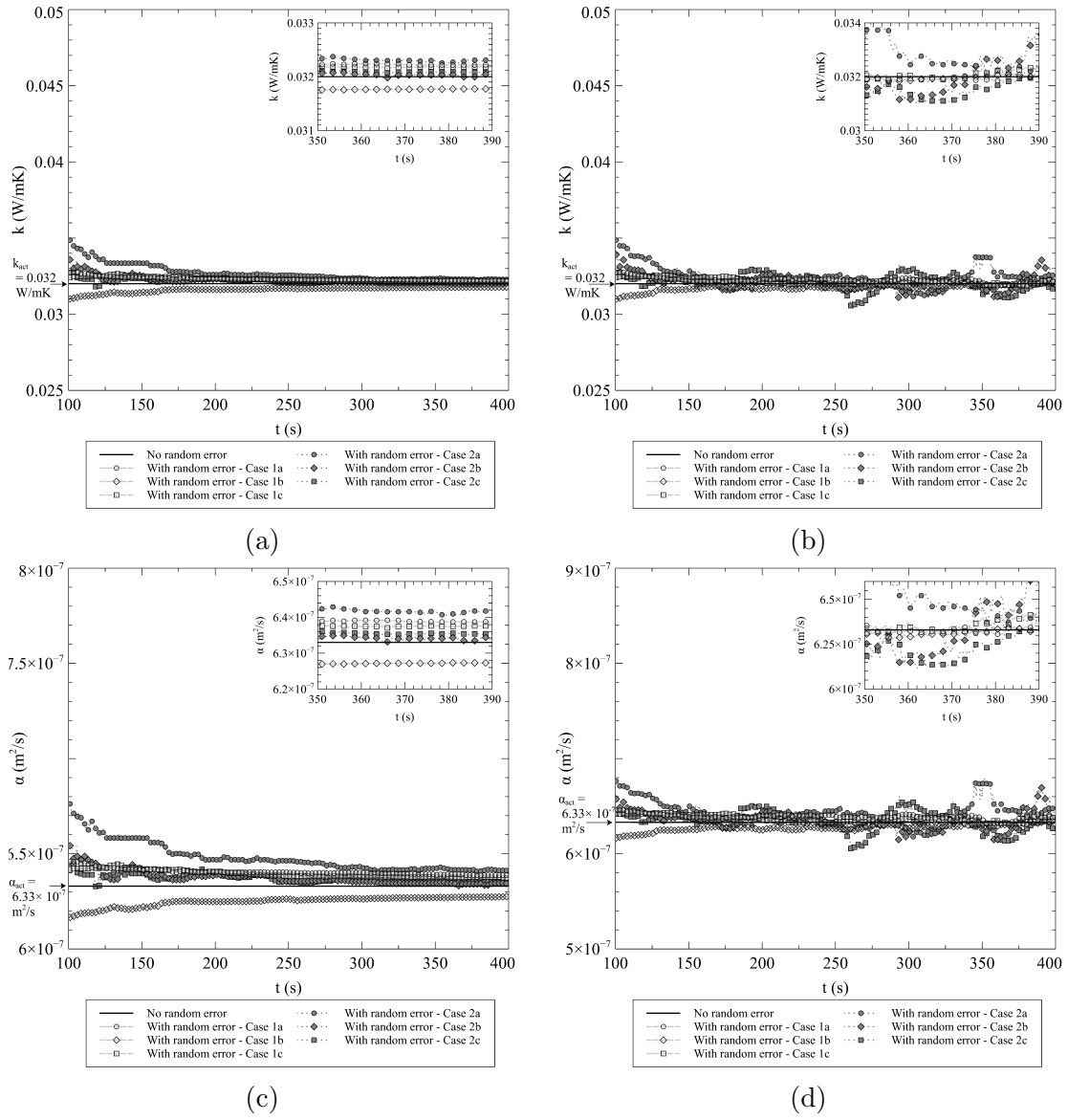

Figure 4: Measurement analysis for XPS: (a) Standard analysis with and without random error compared for estimating thermal conductivity (b) Difference analysis with and without error compared considering time window of 100 s for estimating thermal conductivity (c) Standard analysis with and without random error compared for estimating thermal diffusivity(d) Difference analysis with and without error compared considering time window of 100 s for estimating thermal diffusivity

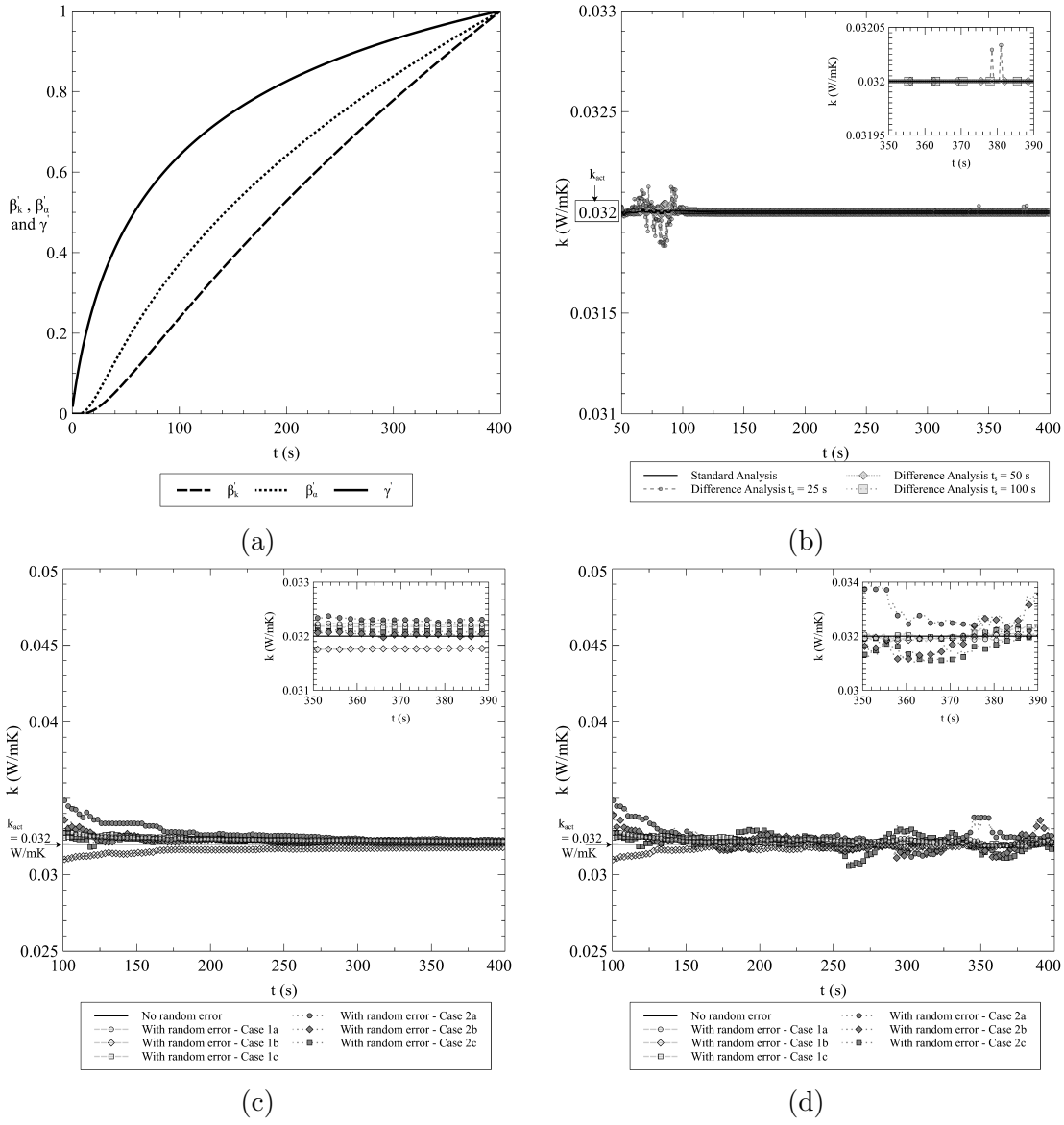

Figure 5: Measurement analysis for XPS: (a) Sensitivity coefficients and their ratio (b) Standard analysis and difference analysis for three different time windows (c) Standard analysis with and without random error compared (d) Difference analysis with and without error compared considering time window of 100 s

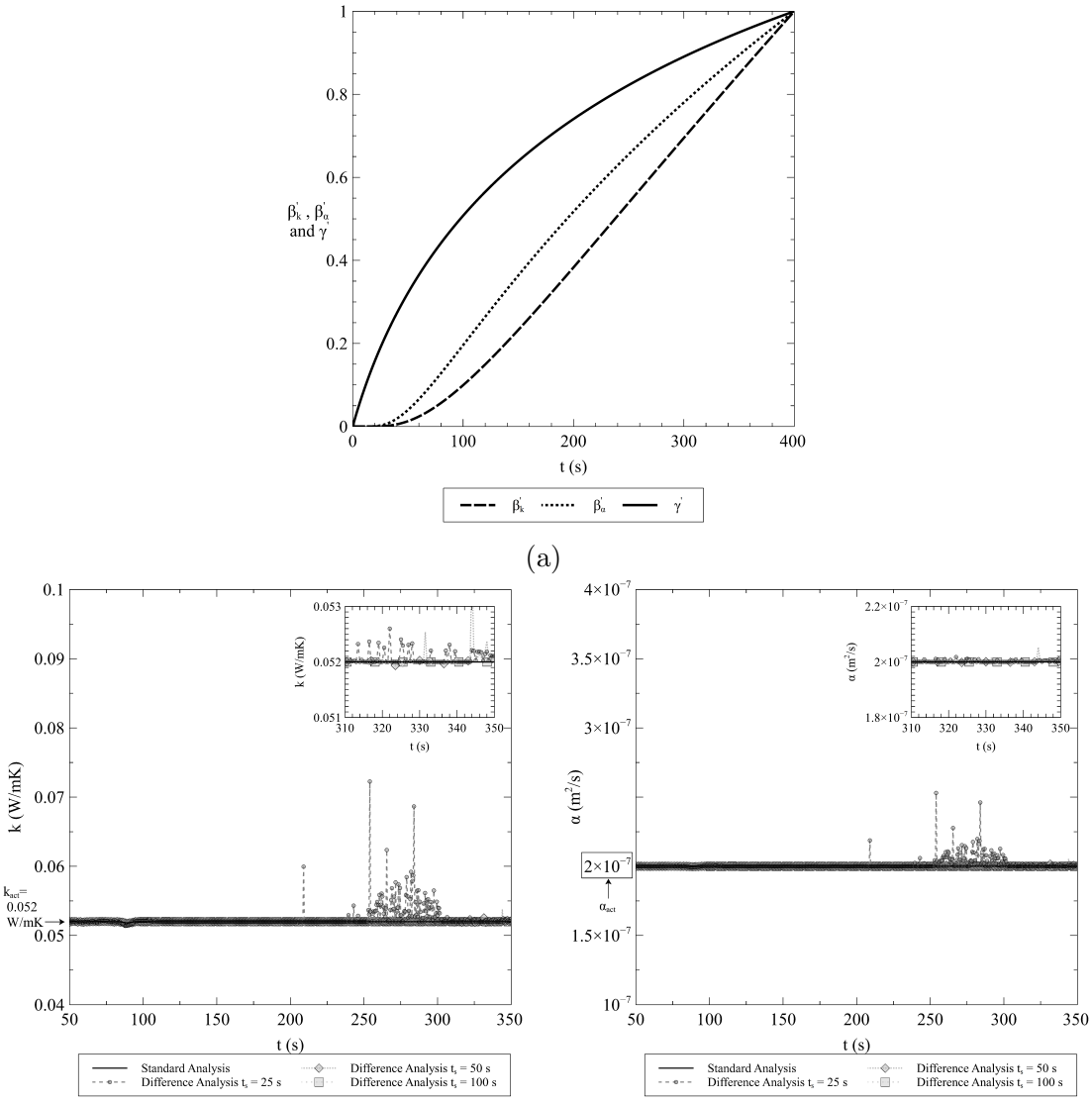

Figure 6: Measurement analysis for Straw: (a) Sensitivity coefficients and their ratio (b) Standard analysis and difference analysis estimating thermal conductivity for three different time windows (c) Standard analysis and difference analysis estimating thermal diffusivity for three different time windows

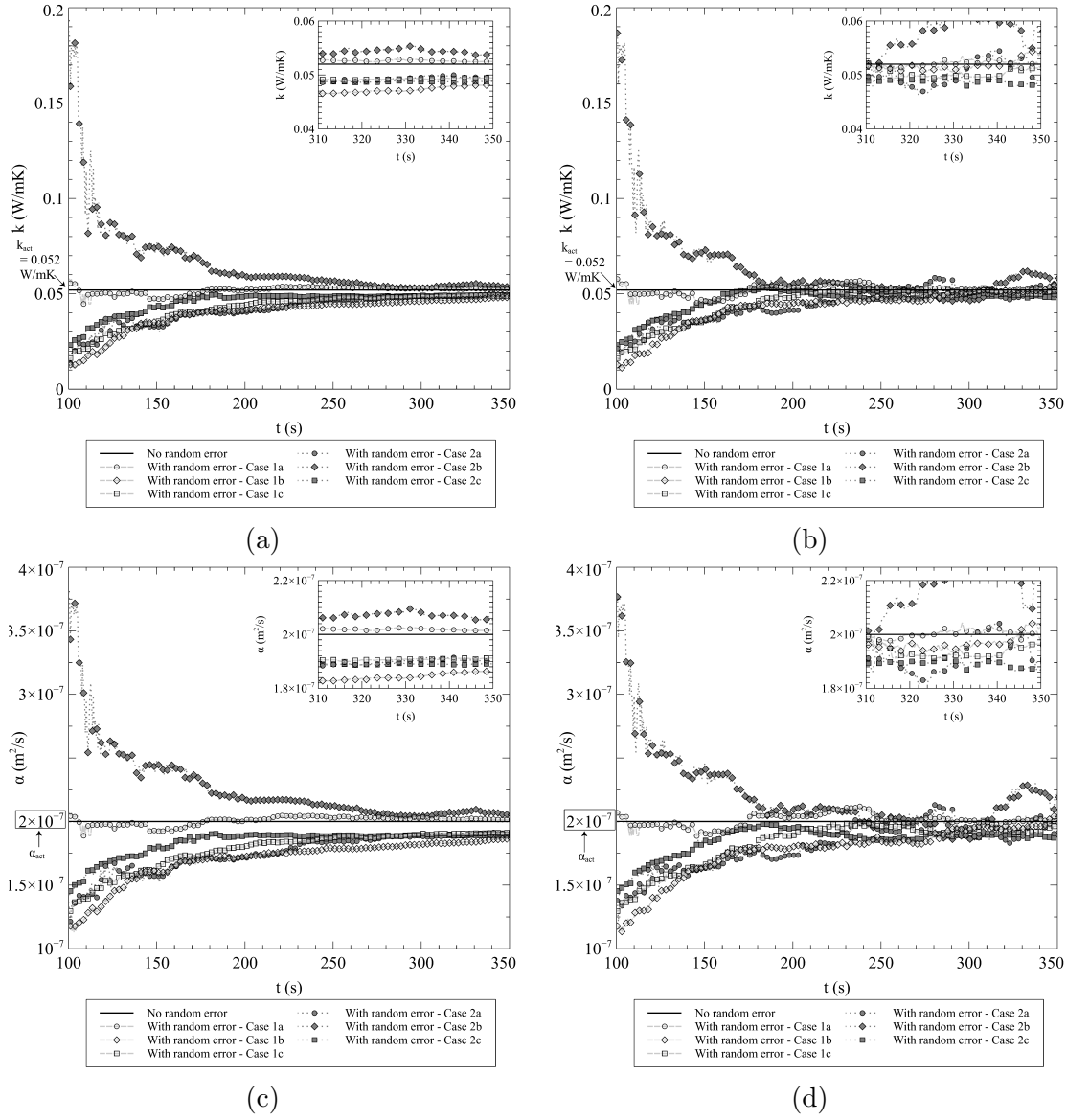

Figure 7: Measurement analysis for Straw: (a) Standard analysis with and without random error compared for estimating thermal conductivity (b) Difference analysis with and without error compared considering time window of 100 s for estimating thermal conductivity (c) Standard analysis with and without random error compared for estimating thermal diffusivity(d) Difference analysis with and without error compared considering time window of 100 s for estimating thermal diffusivity

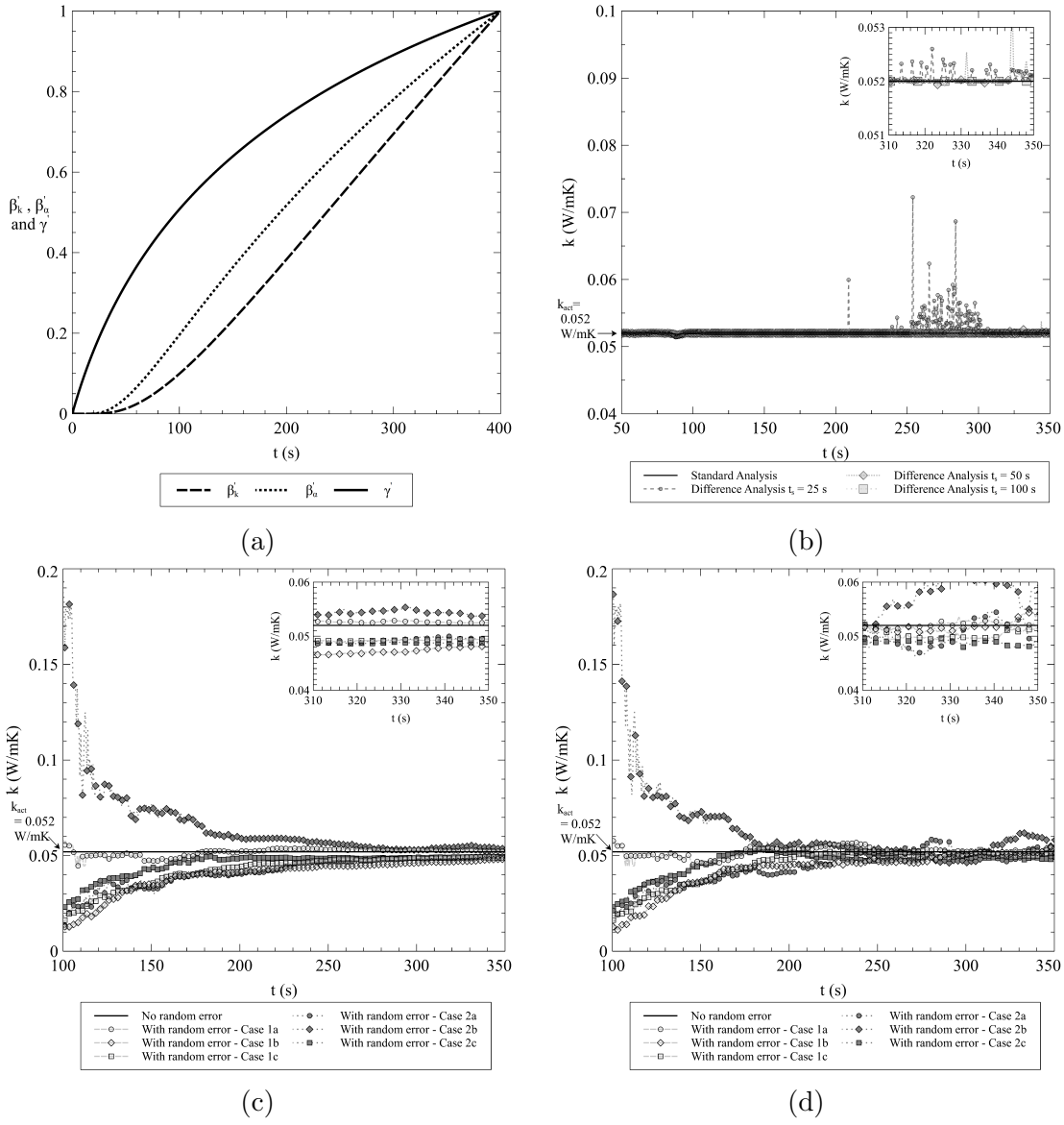

Figure 8: Measurement analysis for Straw: (a) Sensitivity coefficients and their ratio (b) Standard analysis and difference analysis for three different time windows (c) Standard analysis with and without random error compared (d) Difference analysis with and without error compared considering time window of 100 s

	PMMA	XPS	Straw
L (set) $[m]$	0.03	0.081	0.1
W $[m]$	0.10	0.15	0.18
ρ (measured) $[kg/m^3]$	1172.97	39.51	$70 - 130$
$k_{max}[W/mK]$	0.21[Rides]	0.042[Dubois]	0.12 [Chaussinand]
$C_{min}[kJ/kgK]$	-	1280[Al-Ajlan]	1338[Chaussinand]
α_{max} $[m^2/s]$	1.15×10^{-7}[Rides]	8.31×10^{-7}	1.28×10^{-6}
$k_{min}[W/mK]$	0.18[Rides]	0.032[Dubois]	0.052[Chaussinand]
$C_{max}[kJ/kgK]$	-	1280	2000[Chaussinand]
α_{min} $[m^2/s]$	9.0×10^{-8}[Rides]	6.33×10^{-7}	2.0×10^{-7}

Table 1: Parameters considered for measurement analysis for the three different samples