Abstract of PhD Study

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Title: Cavity Ring Down and Laser Induced Breakdown Spectroscopic Techniques for the Study of Toxicants at low Concentration Levels

ABSTRACT

Lasers provided great impetus to optical spectroscopy to probe the microstructure of the matter to great details. The structure of atoms and molecules and their physical properties can be experimentally investigated using different laser spectroscopic techniques. Laser based analytical systems are in use for laboratory investigations of different types of samples in all forms of states (solids, liquids, gases and plasma). In the last few years, two spectroscopic techniques, viz. cavity ring down spectroscopy (CRDS) and laser induced breakdown spectroscopy (LIBS) have emerged as very sensitive and versatile analytical techniques for the investigation and analysis of solids, liquids, plasma and gaseous samples in ultra-low concentrations. Rapid advances in lasers and spectroscopy instrumentation are now leading to the development of compact and portable detection systems for field based analysis of major environmental as well as industrial toxic agents.

The major objective of the research work was to study the toxicants as the traces of gaseous samples and lubricants experimentally by establishing sensitive laser spectroscopic detection techniques such as CRDS and LIBS. Environmental or industrial toxicants like nitrogen dioxide gas (NO_2) and lubricating grease and engine oils were studied using these techniques. There exists a great potential of practical utility for non-intrusive, stand-off as well as in-situ detection and analysis of these major toxicants and locomotive engine lubricants. For the purpose of simpler, compact and less costly experimental set-up the phase shift cavity ring down spectroscopy (PS-CRDS) experiment was set-up using a broadband CW diode laser to detect and study NO_x toxic trace gases in closed CRDS cells. On the basis of the optimization of different cavity parameters like cavity length and laser diode power, a high finesse 80 cm length sealed-off CRD cell was fabricated using mild steel material for conducting the experiments for sensing of NO_2 pre-mixed gas concentrations at trace level. The phase-sensitive cavity ring down spectroscopy PS-CRDS experimental arrangement was

established for the traces detection by optimizing the cavity length between 40 cm to 80 cm at different laser diode powers ranging between 1mW to 100 mW at laser diode modulation. The indigenously designed cavity with cavity length 80 cm and all its components such as vacuum valves, nozzles and mounts were tested at different concentrations of nitrogen dioxide gases in parts per million level concentrations. Experiments were carried out on three pre-mixture of NO_2 gas concentrations taken at 185 ppm, 458 ppm and 590 ppm. The absorption coefficient calculated was of the order 10^{-6} cm^{-1} and limit of detection is estimated to be of the order of ppb level for sensing of NO_2 using the set-up under given conditions. In another experiment of sensitive trace detection, the limit of detection of the PS-CRDS set-up was estimated by diluting the known pre-mixed NO_2 further by introducing dry argon gas in the CRD cell at high pressures. The limit of detection of NO_2 gas in argon environment using this PS-CRDS set-up was determined as 2×10^{11} cm^{-3}.

In the second part of the thesis, laser induced breakdown spectroscopy (LIBS) experiments were set-up for studying the quality of locomotive lubricants, rail engine grease and mobile oil in a standoff detection mode. LIBS experiments were conducted using a Q-switched nanosecond Nd:YAG laser operating at fundamental wavelength 1064 nm and at 10 Hz repetitive frequency. The experimental arrangements with CCD and ICCD spectrometers were installed in order to study the composition of the traces of impurities and degradation quality of the lubricants including engine oils and grease samples. For the present thesis work LIBS experiments were conducted on two types of lubricant samples viz. grease and engine oil and the emission spectra were detected using CCD and ICCD coupled spectrometers. The LIBS spectra were obtained to find out the impurity elements which are present in trace concentrations in lubricant samples. In engine oils, the studies were performed on fresh and dirty engine oils samples and on grease samples, the fresh, partially degraded and fully degraded grease samples which were used to make the comparative studies of the elemental composition. It was analysed that the presence of impurity elements such as barium in engine oil and lead in grease samples can be highly toxic regarding human health and the wear and tear of machinery parts which is an indication to replace the machinery parts. In a much refined experiment with ICCD spectrometer, the existence of several impurity elements such as Si, Fe, Mn, Mg, Ni, Al, Ca, Cu, Pb, S and Zn are the indication of the degradation performance of grease at critical level of the study of grease in terms of its composition structure and utility.