ABSTRACT

Classical analytical procedures are associated with solvent pollution due to large solvent usage, disposal, solvent evaporation and time - consumption. As Montreal protocol insisted on environment friendly analytical procedures, solvent usage has to be reduced. Stockholm convention called for the attention on persistent organic pollutants (POP) and hence poly chlorinated biphenyls (PCBs), penta chloro phenol (PCP), other chlorophenols, hexa chloro hexane and chlorobenzenes which are POPs have been studied. The present study exploited water for the sample extraction employing an open vessel focused microwave extraction technique (FMAE). Of the several modern sample preparation techniques, FMAE has been chosen for this study since it is carried out under normal pressure and it is simple to develop a rapid procedure with minimal solvent usage. Four different approaches were tried for polar to neutral analytes. The first approach involves the extraction of chlorophenols from solid samples using an aqueous carbonate solution in which they swell improving the extractions. The recovery was the best with 0.01 % carbonate at 20% MW power with 10 min duration. The second approach is based on the fact that the MW (microwave) generates heat through a good dielectric like water with which the solid sample is covered. MW heating releases analytes from the hot swollen solid samples, the water insoluble non-polar and volatile analytes rise to the surface where n-octane trap is available to dissolve them. n-octane, a low–dielectric is not MW interactive but picks up heat relayed to it by water.

n-Octane in the range of 1-5 ml was successful for extracting PCBs, chlorobenzenes and γ-HCH from solid matrices and from 25-100ml water samples leading to 5-100 fold enrichment of the analytes. The influential parameters for the recovery of analytes were found out. Spiked recoveries of standards from solid and water samples and real sample analyses were found to exceed 90% with reproducibilities below 5.4% RSD.

The third approach is also based on the same principles as the second, but involves an additional step of insitu acetyl derivatsation of chlorophenols towards an integrated procedural approach also targeted at solvent minimization. Since acetyl derivatisation is needed for GC analysis, the derivatizing agents are added in the extraction pot itself. The study involved optimization of MW conditions for good recoveries. The procedure studied assures complete and quick extraction of analytes, enrichment of 5-100 folds, and along with derivatisation. The recoveries, reproducabilities in real samples are all similar to the second approach.Using both methods, the analytes were extracted in 1-5 min from water samples and 3-15 min from solid samples.

The fourth approach is a rapid one-pot derivatization and distillation of chlorophenols and distillation of PCBs and chloro organics from solid samples and their on-line trapping for enrichment. Microwave assisted steam distillation does not produce large volume of distillate unlike the conventional one for an exhaustive recovery of analytes and produces purified extracts.

This approach also incorporates derivatisation of chlorophenols for better distillability. However other analytes namely PCBs, chlorobenzene compounds and HCH were distilled and trapped on-line using a solid sorbent or

a liquid. MASD has also expanded the scope, offering faster extraction and on-line enrichment of analytes. The study included optimizing MASD for the complete recovery of the analytes with spiked sample studies successfully employed for soil, leather and textile. MASD methods developed are rapid procedure, generating only a few ml of distillates with good recoveries and reproducibilities.