Preliminary application of tapered glass capillary microbeam in MeV-PIXE mapping of longan leaf for elemental concentration distribution analysis

S Natyanun\textsuperscript{1,*}, S Unai\textsuperscript{2}, L D Yu\textsuperscript{3}, U Tippawan\textsuperscript{1} and N Pussadee\textsuperscript{1}

\textsuperscript{1} Plasma and Beam Physics Research Facility, Department of Physics and Materials Science, Faculty of Science, Chiang Mai University, Chiang Mai 50200, Thailand.
\textsuperscript{2} Physics Division, School of Science, University of Phayao, Phayao 56000, Thailand.
\textsuperscript{3} Thailand Center of Excellence in Physics, Commission on Higher Education, 328 Si Ayutthaya Road, Bangkok 10400, Thailand.

* E-mail: bas9sirawut@gmail.com

Abstract. This study was aimed at understanding elemental concentration distribution in local longan leaf for how the plant was affected by the environment or agricultural operation. The analysis applied the MeV-microbeam particle induced X-ray emission (PIXE) mapping technique using a home-developed tapered glass capillary microbeam system at Chiang Mai University. The microbeam was 2-MeV proton beam in 130 µm in diameter. The studying interest was in the difference in the elemental concentrations distributed between the leaf midrib and lamina areas. The micro proton beam analyzed the leaf sample across the leaf midrib edge to the leaf lamina area for total 9 data requisition spots. The resulting data were colored to form a 1D-map of the elemental concentration distribution. Seven dominant elements, Al, S, Cl, K, Ca, Sc and Fe, were identified, the first six of which were found having higher concentrations in the midrib area than in the lamina area, while the Fe concentration was in an opposite trend to that of the others.

1. Introduction
The ion microbeam technology is an extension and also a promotion of general ion beam technology. The main applications of microbeam are (1) processing materials – highly localized and highly controlled ion quantity irradiation of the sample, and (2) analyzing materials – imaging/mapping and probing of the sample such as particle-induced X-ray emission (PIXE) for mapping analysis of samples. Beam spots with a diameter down to about two micrometers can be obtained by collimating the beam with pinhole apertures or with a drawn capillary. We have established the MeV microbeam technology at Chiang Mai University (CMU) with some technical developments for applications in MeV-ion-beam imaging/mapping. Following successful development of the programmable proximity aperture for MeV ion beam lithography, we have been developing another
even cheaper and simpler MeV microbeam technique, namely tapered glass capillary (e.g. [1,2]). In this study, the glass capillary microbeam was utilized for elemental mapping of biological sample. Since local longan trees in Thailand are normally heavily sprayed with fertilizer and pesticide and chemical residues might be long lasted on the leaf, the longan leaf should be analyzed for revealing whether and how the plant was affected by the environment or agricultural operation and hence providing both producers and customers with necessary consultations.

2. Materials and Methods
The CMU MeV microbeam facility is based on our 1.7-MV Tandetron tandem accelerator and its beam line (Fig. 1) which consist of an ion source terminal including two ion sources, a duoplasmatron ion source and a Cs sputter ion source, a switching magnet with two entrances and one exit, the accelerator including the high-voltage supply, the post-acceleration beam transportation line including a 30° mass analyzing magnet and quadrupole lenses, and an endstation chamber. The end-station chamber is mainly applied for ion beam analysis.

The tapered glass microcapillary system [3] consists of a 0.8-mm beam collimator to guide the beam into the capillary tube, the glass tapered capillary tube fixed in an Al frame, and a rotator for moving the glass tube away or into the beam direction. The sample stage is X-Y moveable with a translation repeatability of 4 µm and an accuracy of 6 µm. The glass capillary in different sizes is changeable to install in the capillary holder. After each time of the installation, beam alignment is firstly carried out with the help of using a laser beam. A laser source is installed under the beam line just in the front of the endstation and the laser beam is deflected by a rotatable mirror inside the beam line to direct to the capillary. Simple manual adjustment of the capillary can finally achieve a satisfying alignment by looking at the best laser beam spot at the target position through the capillary.

The tapered glass microcapillary system was installed within a four-columns frame which was then put inside the endstation, applied for MeV-microbeam PIXE mapping to reveal the elemental concentration distribution in local longan leaf, especially the difference between the leaf midrib and lamina areas. In the analysis, a fresh longan leaf was first dried in an oven at 80°C for 2 hours, and then cut into sample with a size of 6 mm by 8 mm, particularly including midrib and lamina areas, as shown in Fig 2. The sample were fixed on the sample holder, which was then put on the sample stage (Fig. 3) in the analysis chamber under a pressure of 10⁻⁴ Pa. The microbeam was coming from the capillary with about 100 µm in inner diameter of the exit and the ion beam was of 2-MeV proton. SRIM simulation showed the range of 2-MeV proton in biological tissue equivalent polymer materials to be 65 µm, while the thicknesses of both leaf midrib and lamina cross sections were much greater than this range (measured thickness of the midrib about 500 µm and the lamina about 200-300 µm, as shown in Fig. 4), and therefore the proton beam evenly analyzed every part of the entire leaf materials. The micro proton beam analyzed the leaf sample at a fixed spot of about 130 µm in diameter for 1000 seconds, while the leaf held on the sample stage was translated step by step over a 1,350-µm range across the leaf midrib edge to the leaf lamina area, resulting in total 9 data requisition spots to realize a scanning of the interesting area.

![Figure 1. Schematic diagram of the beam line of the 1.7-MV Tandetron accelerator and beam line at Chiang Mai University. The beam line length is about 10 m.](image-url)
3. Results and Discussion
From nine PIXE spectra, the X-ray peak heights were converted to corresponding elemental concentrations, which were then normalized for all of the elements detected. Totally 7 elements were identified in dominance. The resulting data were colored to form a 1-D map of the elemental concentration distribution, as shown in Fig. 5. It was interestingly found that in the midrib region, the concentrations of Al, S, Cl, K, Ca, and Sc were relatively higher than in the lamina area, whereas the Fe concentration was in an opposite trend to that of the others. The only indication of fertilizer and pesticide elements detected in the leaf sample was of K, which had the relatively higher concentration in the midrib compared to other elements. Due to a higher mass density of the leaf midrib compared with the lamina, it should be natural that the elemental concentrations are normally higher in
the midrib parts than in the lamina parts. For the case of Fe, it is speculated that the leaf lamina can much more easily concentrate this element than the midrib, probably owing to the lamina structure providing easy locations for Fe to segregate.

Figure 5. MeV microbeam mapping of the midrib edge part of a local longan leaf. (a) A microphotograph of the analyzed leaf part. The white box indicates the PIXE mapping area, where the numbers indicate the position in µm of each microbeam-analyzed spot. (b) Elemental maps (in 1D) of the midrib-lamina part (midrib at right side and lamina at left side, in the same direction in (a))

4. Conclusions
Economic tapered glass capillary microbeam technology has been home-developed at Chiang Mai University. The capillary system is equipped with an in-house-made tapered glass capillary with a µm-exit. Cooperating with X-Y translations of the sample stage, the system allows microbeam scanning possible. In the preliminary application, local longan leaf was scanned by the MeV proton microbeam for elemental mapping to investigate possible contamination from fertilizer and pesticide applications. Seven dominant elements, Al, S, Cl, K, Ca, Sc and Fe, were detected and their elemental concentration distributions were mapped in one dimension. Most of the elements were distributed in the midrib part more than in the lamina part, except Fe which had an opposite distribution trend to the others.

5. References
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