Acid-Base Interactions:

Relevance to Adhesion Science and Technology

Editors
K.L. Mittal and H.R. Anderson, Jr.

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In Honor of the 75th Birthday
of Professor Frederick M. Fowkes

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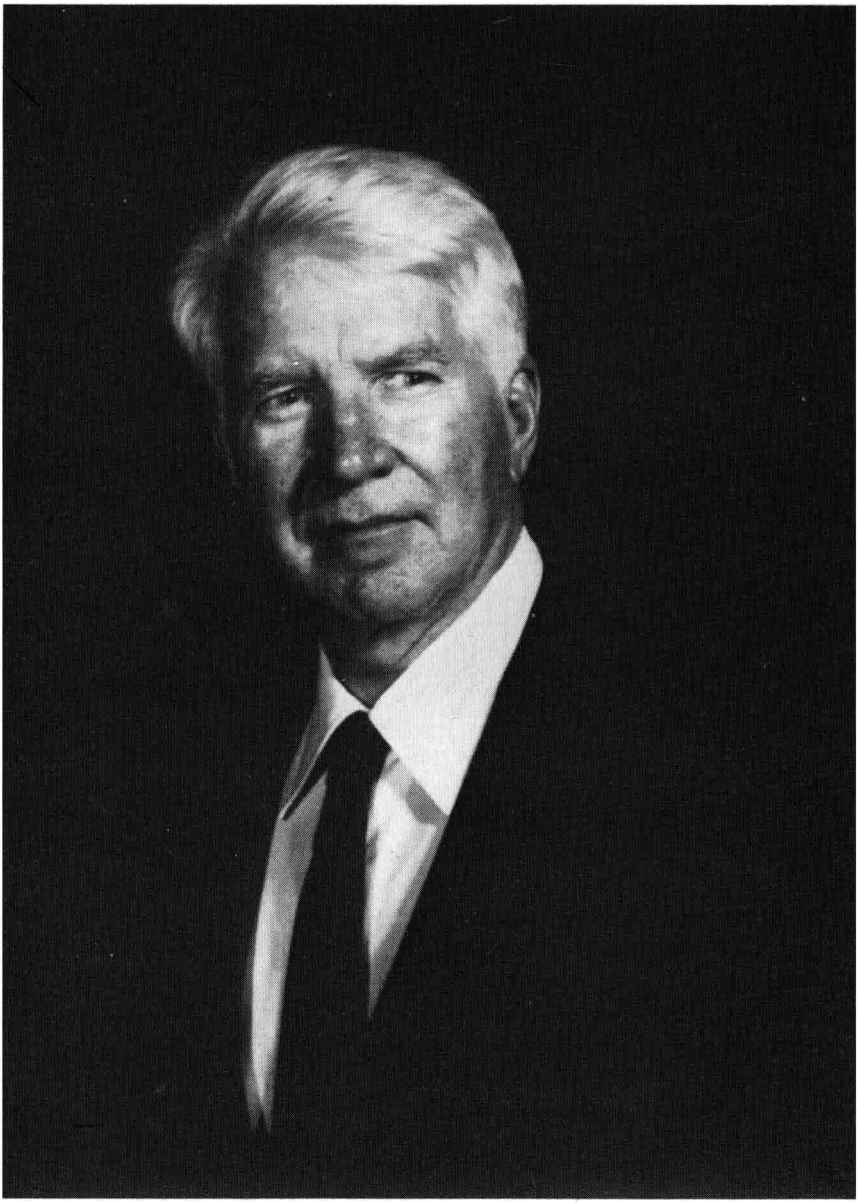

Frederick M. Fowkes (1915-1990)

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Eds. K.L. Mittal and H.R. Anderson, Jr.

Preface

This book chronicles the Proceedings of the Symposium on Acid–Base Interactions: Relevance to Adhesion Science and Technology held on the occasion of the 75th birthday of Professor Frederick M. Fowkes as a part of the 64th Colloid and Surface Science Symposium held at Lehigh University, June 18–20, 1990. As a matter of fact the papers from this symposium were earlier published in four issues of the *Journal of Adhesion Science and Technology* (*JAST*) as follows: Vol. 4, No. 4 (1990); Vol. 4, No. 5 (1990); Vol. 4, No. 8 (1990); and Vol. 5, No. 1 (1991) which were dedicated to him as a Festschrift in his honor. However, a large number of people indicated interest in acquiring these four issues separately; concomitantly, we decided to bring out this special commemorative volume. But as most of you may already be aware, Prof. Fowkes passed away on October 18, 1990 and the present volume now represents a memorial to this 'giant' of adhesion science. Death came to him suddenly, but painlessly; and he was quite active till the last moment.

When the idea of organizing a symposium to honor Dr. Fowkes was conceived, the question arose as to what should be the theme of the symposium as he had made significant contributions dealing with the many ramifications of interfacial science and technology. However, after some deliberation, it became clear that a most apposite topic would be 'acid–base and adhesion', as in the last several years the leitmotif of Dr. Fowkes's research has been in the realm of acid–base interactions. So it was decided to hold the symposium under the rubric 'Acid–Base Interactions: Relevance to Adhesion Science and Technology'. The contributions to the Symposium were mostly by invitation only, and there was a consensus among those invited that this event was very opportune to honor this doyen of adhesion science.

Here, we will not expatiate upon the many and significant contributions of Dr. Fowkes as these can be easily gathered from the following excellent write-up by Dr. Ross. However, we would like to reinforce that Dr. Fowkes had pioneered many ideas, and the surface and colloid community owes much gratitude and appreciation to him for his research efforts spanning over half a century. In the last few years, he had been a key protagonist of the concepts of acid–base interactions, had done a lot to popularize such concepts, and had shown how understanding of such interactions could be parlayed to control adhesion behavior in a given situation. As a result of his pioneering efforts there has been a paradigm shift in our approach to adhesion science in terms of acid–base interactions. By the way, these days the phrase 'paradigm shift' is much in vogue in the business and technological world, and we adhesionists have seen our own paradigm shift

(from polar–polar interactions to acid–base interactions) and much credit goes to Dr. Fowkes for this transformation.

Now turning to the present volume, here the papers have been rearranged in a more logical manner *vis-à-vis* the order in which they were published in the special issues of *JAST* (*vide supra*). The book opens with an excellent write-up summarizing the professional and personal life of Dr. Fowkes by Dr. Sydney Ross. The rest of the text containing 22 papers is divided into three parts as follows: Part I. Fundamental Aspects of Acid–Base Interactions; Part II. Characterization of the Acid–Base Properties of Materials; and Part III. Applications of Acid–Base Interactions. Among the topics covered include: Acid–base concepts: historical account, current status, and prospects for the future; quantum-mechanical approach to understanding acid–base interactions at metal–polymer interfaces; assessment of acid–base interactions at solid–liquid interfaces; quantitative characterization of the acid–base properties of solvents, polymers, and inorganic surfaces (an overview by Prof. Fowkes himself); acid–base characteristics of a variety of solid materials (clay minerals, carbon fibers, glass fibers, silicas, metals, polymers); acid–base interactions in wetting; and applications of acid–base interactions in a variety of situations, e.g. in the adhesion of polymers to metallic and ceramic substrates, mechanical properties of wood, properties of filled polymers, and behavior of fiber-reinforced polymer composites.

In essence, this volume represents the cumulative wisdom and thinking of contemporary researchers engaged actively in unravelling acid–base interactions and how these can be utilized in understanding and tailoring adhesion. Furthermore, we certainly hope this compendium will become the *vade macum* for anyone interested in acid–base interactions and adhesion. The information provided in this book should serve as a fountain of new ideas for researchers in adhesion to, hopefully, make a quantum leap in understanding the phenomenon of adhesion and concomitantly contribute substantially to the wonderful world of adhesion science.

In closing, we would like to add that this book should serve as a constant reminder of Prof. Fowkes's seminal contributions anent acid–base interactions and adhesion. Needless to say, he will be sorely missed by adhesionists world wide and particularly by those who had the good fortune to come in contact with this gentle soul and to learn from this maestro of adhesion science.

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Frederick M. Fowkes: Industrial and academic scientist

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When Dr Mittal invited me to write a personal account of Professor Fowkes for a symposium to be held in his honor and in commemoration of his seventy-fifth birthday, I was pleased at the opportunity to present his interesting life story and the details of his productive career. I am an old friend and contemporary of the professor and yield to none in my appreciation of his contributions to science as well as in my esteem for the higher faculties of his mind and the higher qualities of his character, the former summed up as intelligence and the latter as integrity. His intelligence may be relied on for dispassionate judgments; and his integrity for a sincerity that does not always stop short of candor. (This last trait is not welcomed in all quarters.)

Frederick Mayhew Fowkes was born on January 29, 1915 in Chicago, Illinois. His father, William Herbert Fowkes, was an English-born patent lawyer who after graduating from Barnard Castle, a public school (in the English sense) in County Durham, saw action with the Northumberland Fusiliers in the Boer War, served in the Yukon with the Canadian Northwest Mounted Police and then served five years with the U.S. Army in the Philippine Insurrection before attending the Chicago–Kent College of Law. His mother, Eleanor Mayhew (Seley) Fowkes, came from a family of English heritage, which had emigrated to Minnesota and Illinois in the mid-nineteenth century. She was a graduate of the University of Chicago, with a degree in botany.

Fred Fowkes was raised in suburban Chicago and was educated in the public school system of that city. His father died in the flu epidemic of 1919–20 and afterwards his maternal grandparents lived with the family. His grandfather, Charles Arthur Seley, was an inventor and the chief mechanical engineer of the Norfolk and Western Railroad and later of the Rock Island Railroad. He loved music and engineering, and evidently became a role model to his grandson. He continued to work as a design engineer into his eighties. Fred and his brother Bob grew up in a household filled with interest in church (especially choir) music, in instrumental music, in athletics, and in science and electronics. Fred played piano, played the tuba in symphony orchestras, in marching bands, and even in dance bands. In high school he became the tuba player for the All-Chicago High School Symphony Orchestra, and actually played six concerts with the Chicago Symphony Orchestra.

He attended Morgan Park High School, which was more than fifteen miles from the center of the city in a suburban-type neighborhood, of upper-middle class

background. It was a small school by Chicago standards and a large fraction of its graduates went on to college. He found the chemistry class uninteresting but he still remembers the good teaching in mathematics, biology and physics. He played on the football team every fall, failing typing as a result of breaking his right elbow in wrestling, and failing it again after tearing ligaments in his left wrist in football. He took about thirty credits more than was required to graduate.

Fowkes became an undergraduate at the University of Chicago at the time when its president Robert Maynard Hutchins (1899–1977) put into effect his exciting 'new plan', with its emphasis on a broad education, on reading the 'great books', on student contact with outstanding professors, and on much personal freedom for students. Class attendance was not required and grades were based only on final exams, consequently students were encouraged to take heavy course loads. Fowkes completed all his undergraduate course work for the bachelor's degree in Chemistry within 2 years, and started taking graduate courses and doing graduate research with William Draper Harkins (1873–1951) in 1934. In spite of his heavy work load he managed to play on the football team during his first year, to play the tuba in the University Symphony Orchestra, and to be an active member of Phi Gamma Delta fraternity, where he became a close friend of Luis W. Alvarez (1911–1988; Nobel laureate in physics, 1968).

To work in Harkins' laboratory was to be introduced to a stimulating research environment, because of the presence of many outstanding post-doctoral fellows and because of the variety of subjects under study, which included nuclear reactions. Frank Long was Fowkes' lab-mate for several years, and both of them accompanied Harkins for a semester at Cornell University during 1936–37. Long eventually became a well-known professor at Cornell.

Fowkes' first surface-chemical studies concerned two-component monolayers (fatty acids plus hydrocarbons). By means of an ultramicroscope that he built into the film balance, he observed the ejection of the oil from the mixed monolayer upon compression [1].* His Ph.D. dissertation was a study of monolayers adsorbed from aqueous solutions by various hydrophobic materials. The pressure-area relations were determined by simultaneous measurements of surface tension and contact angles [3]. This was the first of many subsequent studies of contact angles in Harkins' laboratory, and as a pioneer it had been up to Fowkes to build the equipment himself.

His first employment was a 6-month stint at the research laboratories of Nalco, while he was still a graduate student. He continued his University research during evenings and weekends. Then he was awarded a Proctor and Gamble fellowship, a rare opportunity in those years of the Great Depression. He gave up his job at Nalco, and completed all his thesis research in the next year. Meanwhile he had married Royce Elizabeth Budge, whom he had met when he was a graduate student and she was an art student at the Art Institute of Chicago. A friend in common had brought them together at a picnic meeting of Episcopalian students at the University. Their four children are Gordon Seley (born May 11, 1939), Joan Berkeley *Piper* (born December 19, 1941), Mary-Elisabeth *Tobin* (born January 18, 1950) and Virginia Mayhew *Clark* (born September 19, 1958).

*Numbers within brackets refer to items in the numbered List of Publications of F. M. Fowkes appended to this memoir.

Fowkes obtained the Ph.D degree in 1938 (still in the depression period) and went to work in the research laboratories of the Continental Can Company in Chicago, where he was put in charge of a group testing the interaction of polymeric can linings with all the various foods that are canned. He had asked to study the effects of foods on the iron/tin electric couple, for it was quickly evident that tin was the anode in citrus juice, but iron was the anode in canned cherries, strawberries, plums, etc.; but he was always required to put the testing of polymer coatings first, and though his group expanded to seven people, there was never time for fundamental research. So when he was called to serve in the Army in 1942, he knew he did not wish to return to his job at Continental Can.

Fowkes had been in the Reserve Officers Training Corps (ROTC) in college, and had accepted duty as a reserve officer attached to the regular army Third Field Artillery at Fort Sheridan, Illinois. However, when he was called up for active duty as a first lieutenant in February 1942, he was sent to Fort Sill, Oklahoma, together with about forty other artillery reserve officers, for a training class. Such classes started each week and continued for eight weeks. His class included about five chemists with Ph.D. degrees; it also included Temple Fielding, who later wrote the well known Fielding's Guides to Europe.

At Fort Sill Fowkes taught recruits to fire 155-mm howitzers and to drive huge Diamond T trucks. He was soon promoted to captain and put in charge of field exercises for a 10 000-man replacement training center, his task being to schedule all artillery practice and to conduct gas-mask drills. In June 1943 he was transferred to the 31st (Dixie) Infantry Division just as it was going into summer maneuvers in Louisiana, and in September he was assigned to command Battery C of the 149th Field Artillery, a 105-mm howitzer unit in support of the 124th Infantry Regiment. They trained in Virginia, where, he says, "I learned how to motivate troops. I found that all I had to do was to tell the men each morning exactly what was going on, and just what they were expected to do. Once the men knew this, they did their best. We developed high morale and efficiency." They were sent in January 1943 by ship from Virginia to the Panama Canal with a British naval convoy; from there to New Guinea they were unescorted.

After acclimatization in New Guinea, they were sent by landing craft to Aitape, New Guinea, to help defend troops there from a much larger force of attacking Japanese. They restored the broken front lines and experienced tough combat, for their immediate foe was the picked division of 6-foot Japanese who had conquered Singapore. The Americans had expected less formidable opposition and were shocked. But though their gun positions were often attacked at night, and once one of their ammunition dumps was exploded, they did a good job of defending the Drinamour River line, and turned back the attack with great losses to the enemy.

In September 1943 the 31st Division was sent by landing craft to Moratai, a small island in the Spice Islands of the Netherlands East Indies (now Indonesia). General MacArthur walked ashore with them at their landing and inspected the air field, which was soon made into a major air base for all the land-based bombing of the Philippines and even of Borneo. They, in turn, were bombed and strafed 82 nights by the Japanese.

In April 1944 Fowkes was sent to Leyte by plane, to go along with the landing of the 24th Infantry Division in Mindanao, the big southern island of the Philippines.

The 31st Division soon joined them and they were transported by boats a hundred miles up the Mindanao River, where they immediately got into heavy fighting as they drove northwards along the Sayre Highway. They were ambushed frequently and lost many men. Fowkes' assignment was as a liaison officer with an infantry battalion, which, being the most aggressive, was put into the lead and lost the most men. It was finally relieved of the lead when casualties had reduced its strength from 1000 to 245. After one tough attack in which they were subjected to a Banzai charge that splintered the battalion, Fowkes gained the Silver Star medal for Gallantry in Action: he had taken command of the remnants of five infantry companies, organized them, and led them out of danger to rejoin the rest of the battalion.

After this action the 31st went into training for the invasion of Honshu. They were to land east of Tokyo and fight their way into the city. Fortunately the atomic bomb ended the war before such a landing was necessary. By now Fowkes had the rank of major. He did not get back to the States (San Francisco) until December 19, 1945. While waiting for trains east he lived aboard ship at the Embarcadero and looked for a job in chemical research in the Bay Area. When he visited the Shell Development Company (wearing a wool uniform issued to him from a barrel on the dock), Dr Tamale, head of the Catalysis and Surface Chemistry Department, pulled out of his files a reprint of Fowkes' 1940 paper with Harkins. No further recommendation was required; he was immediately offered a job as Research Supervisor.

The Shell Development Company (of Emoryville, California; close to Berkeley) in the forties and fifties was an exciting and advanced research facility. There were about 450 scientists with Ph.D. degrees plus about another 800 people in support. It was the largest research laboratory on the west coast. There was much emphasis on buying or building the best and most advanced laboratory equipment. Faculty members from Berkeley and Stanford often came to use instruments that they yet lacked, such as an NMR spectrometer or an electron microscope. There was also much emphasis on hiring the most innovative people, who came from many countries and institutions. Fowkes' immediate boss had been the first graduate student of Jaroslav Heyrovski (1890–1967), and his boss's boss was from Holland. Fowkes was given the opportunity to build up a well instrumented surface-chemical laboratory, designing many instruments himself; and he was assigned several able researchers, such as Martin Schick and Webster Sawyer.

Fowkes' research group concentrated on the understanding of surface-active solutes, both water-soluble and oil-soluble, with emphasis on the latter, of course. They pioneered the understanding of non-aqueous micelles, of oil-soluble surface-active polymers, of electrostatic and steric stabilization in organic media, of surface-active cosolutes for aqueous systems, of the mechanism of antifoaming additives, and the concept of dispersion forces and acid–base contributions to the work of adhesion and to the surface tension of liquids and solids. The work was a fifty–fifty balance of fundamental and applied studies, in which each part supported the other. The applied problems included gasoline additives, lubricating oil additives, clay-based greases, emulsification processes for crude oils, antifoams for hydrocarbon systems, formulations of aqueous surface-active solutes, anti-corrosion formulations, studies of molecular-weight distributions of epoxies and elastomers, Ziegler catalysis within micelles for elastomer synthesis, etc. Most

of this work was approved for publication, though an exception that Fowkes much regretted was the work he did in Amsterdam in 1955–56, when he studied the acidity and basicity of the polar cores of non-aqueous micelles, using indicators to determine H_0 (the Hammett acidity function) of the micellar cores. About 1960, he and his collaborators were publishing about 1% of the articles in the *Journal of Physical Chemistry*. It is clear that Fowkes owes the direction of his subsequent career to the opportunities he received at the Shell Development Company.

In 1962 the Sprague Electric Company (North Adams, Massachusetts) offered Fowkes the position of Director of Research in a new Research Center, for which he was ultimately to recruit about half of its eventual 200 people, 45 of whom had Ph.D. degrees, most of them in physics. The research was at the cutting edge of the new semiconductor and integrated-circuit business. With Fowkes came two others from Shell Development: Frank Anderson (now with IBM) and Kenneth Manchester. Manchester went on to develop the idea of ion implantation in semiconductors, bringing it into plant operation, for the first time anywhere, in the late 1960s. Under Fowkes' direction the Sprague laboratory pioneered many other developments that are now key techniques of the semiconductor industry, such as plasma-assisted chemical deposition of silicon oxides, nitrides, and carbides. Fowkes' personal research included: (a) a study of the distribution of sodium ions or protons in the surface region of silicon dioxide or silicon nitride insulators, which proved to have a perfect Poisson–Boltzmann distribution of sodium counterions in the oxide, balancing the SiO^- surface charges [37]; and (b) a demonstration of the enhancement of mechanical properties of composite dielectrics resulting from the modification of the high dielectric-constant filler to provide the surface acidity needed to interact with a basic polymer matrix.

Fowkes was disappointed to find that Sprague Electric was too timid to introduce the new products and processes developed in their R&D Center. When sales declined in the late 1960s, he was ordered to lay off his fine research staff. He resigned in 1968 and accepted an offer to chair the Chemistry Department at Lehigh University. The lay-off at Sprague Electric continued; finally only about twenty people were left in the R&D Center.

As chairman of the Chemistry Department at Lehigh, Fowkes arrived in time to superintend the planning and construction of a new building, which required political scheming as well as much work with architects. By 1975 the Department moved into the $7 million S.G. Mudd Chemistry Building with attached Neville Auditorium, a modern facility with the best of laboratory furniture, hoods, and ventilation, and with a modern mirror-glass facade. Fowkes' goal as chairman was to transform a teaching faculty into a more research-oriented faculty. His style as a leader was to ask advice from professors but to make all the decisions (and to take all the blame) himself. During his 13 years as Chairman the research funding in Chemistry grew to about $2.5 million annually; it has nearly doubled since then.

During his years as Chairman his research program was small, for the job took about 60% of his time. After his retirement in 1981 from the duties of the Chair he continued to teach one course each semester, and he had much more time to write proposals and manuscripts for publication. His 'retirement' started by his becoming a Visiting Scientist at the Materials Laboratory of Wright–Patterson Air Force Base (WPAFB) near Dayton, Ohio. For 2 years during the Fall and Spring semesters at Lehigh he spent every Monday and Tuesday at WPAFB, driving or

flying back for his Wednesday-afternoon class. In the summers he spent longer periods at WPAFB, where he had a two- to five-man research group, an FTIR spectrometer to study chemical shifts associated with polymer–solvent interactions, and a Cahn microbalance to measure the swelling of polymers by solvents. He had proposed to determine the acid–base characteristics of polymer–solvent interactions, which work was successfully concluded with publication of "Acid–Base complexes of polymers" [78].

During the last few years Fowkes' research group at Lehigh has averaged ten to fifteen members, with much help from Dr. T. B. Lloyd (a Research Scientist), Dr. D. W. Dwight (an Adjunct Professor), and Dr. David A. Cole (a postdoctoral fellow). He has received research grants from ONR, NSF, and DOE, but most of his support has come from industry. At present Fowkes has a DOE grant and about eight industrial grants supporting seven graduate students, four post-doctoral fellows, Lloyd, Dwight, himself and his secretary Dawne Kressler.

Fowkes came to Lehigh with 40 publications from his 27 years in industrial research. At Lehigh there have been about 65 additional publications. His early work at Lehigh was in solid-state chemistry, but soon the work reverted to colloid and surface chemistry.

On revient toujours
A ses premiers amours.

Nowadays most of his studies concern molecular interactions in solution, with emphasis on spectroscopic methods of measuring molecular interactions. His first Ph.D. student at Lehigh was Dennis Hess, who is now a professor of chemical engineering at Berkeley. Since Hess, ten other of his students have received the Ph.D. degree, and by the end of the 1989–90 academic year four others will finish, totalling fifteen. Eight other students have taken their M.S. degree with him, including Professor Cesar Silebi of Lehigh's Chemical Engineering Department.

Fowkes' other professional activities included numerous consultancies. He was a consultant to NSF on materials laboratories, to NIH on dental and pulmonary research, to NBS on polymers, to Redstone Arsenal on composite propellants, to Lawrence Livermore Laboratories and Idaho National Laboratory on flocculation in geothermal-energy recovery. His industrial consultancies include those with the 3M Company (12 years,) the Shell Development Company, the Union Carbon and Carbide Corporation, the Atlas Powder Company (now ICI Americas), duPont de Nemours and Company, etc. Fowkes has also been active in various scientific societies. He is a member of ACS (since 1935), the Electrochemical Society, IEEE, and IRI (Industrial Research Institute). He was the Chairman of the ACS Division of Colloid and Surface Chemistry in 1968, he has chaired two Gordon Research Conferences (Chemistry at Interfaces, 1971; and Science of Adhesion, 1973). The professional honors that he has received include the Hillman Award of Lehigh University for the "person who has done the most for Lehigh University", 1979. A Symposium in his honor, sponsored by the Division of Colloid and Surface Chemistry of the ACS, was held at the Colloid and Surface Science Symposium at Lehigh University in 1980. The present occasion is another such Symposium, ten years later, with a large program of invited papers under the chairmanship of Dr K. L. Mittal. One of his papers [25] earned the appellation 'Citation Classic' on May 5, 1980, having been cited over 195 times in the

chemical literature. In 1989 he received the Adhesion Society Award for Excellence in Adhesion Science, sponsored by 3M.

My own association with Professor Fowkes began when he was at Sprague Electric and resided at Williamstown, Massachusetts, not far from Troy, New York. He was an adjunct professor at RPI, teaching a night course on surface and colloid chemistry. He joined me in 1967 in teaching one of the short courses of the American Chemical Society, entitled "Emulsions and Dispersions". For several years this course was given four or five times a year, but a declining economy and a growing competition have since reduced it to only once or twice a year. At present it is a 4-day course on colloid and surface chemistry applied to industrial R&D, sponsored by RPI. As teachers Fowkes and I have been joined by my former student Dr Ian D. Morrison of the Xerox Corporation. Over the 20 or so years that the course has been taught it has acquired about 2000 alumni, and has resulted in a textbook *Colloidal Systems and Interfaces*, by S. Ross and I. D. Morrison. (John Wiley and Sons, 1988.) Although Fowkes was too busy to be a co-author, this book bears the imprint of his teaching, as its authors had a unique opportunity to hear repeatedly lessons from the master.

Fowkes' experience illustrates how much benefit can be conferred on a University by taking into its faculty a mature industrial scientist of demonstrated productivity. Such a policy, like the quality of mercy, is twice blessed: "it blesseth him that gives, and him that takes". In the course of his career in industry, Fowkes discovered generalities of behavior that, later, in a University laboratory, with the aid of post-doctoral fellows and graduate students, he was able to test with purified and representative compounds. Such a professor has a research program already in mind, with more than an inkling of how it is going to come out. That knowledge is a great advantage in picking topics for Ph.D. dissertations, as he has a shrewd idea that positive results can be obtained within a limited time period. It is also useful in writing proposals for funding with confidence and with a first-hand appreciation of the ultimate significance of the work.

With so distinguished a career behind him, Professor Fowkes might well spend his remaining days in well earned leisure, but I hardly expect such an outcome, for he reverses an old Scottish proverb and believes, as well as sometimes practices, that it is better to work for nothing than to sit idle. But then there is another proverb, ascribed to St Jerome, that perhaps lies nearer the mark: Keep doing some kind of work, that the devil may not find you unemployed. We have the authority of the Hymnal that idle hands attract Satan, and for the kind of work that he then puts them to. With these awesome injunctions, which anyway he shows every sign of heeding. I wish Professor Fowkes godspeed in his undertakings, and many more years of unmischievous, even if ungainful, employment.

PUBLICATION LIST OF DR. F. M. FOWKES

Books

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