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Shelf Life Estimation for Water based Paints with Regression Methods.

Obidi Olayide F[1]*, Nwachukwu Simon C[1], Aboaba Olusimbo O[1], and Nwalor John U[2]

[1]Department of Botany and Microbiology, University of Lagos, Lagos - 11001, Nigeria.
[2]Department of Chemical Engineering, University of Lagos, Lagos - 11001, Nigeria.

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***For Correspondence:**

Department of Botany and Microbiology, University of Lagos, Lagos - 11001, Nigeria.
Phone: +234 803 472 0933

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ABSTRACT

The shelf lives of water-based paints made in Nigeria were investigated. The mean changes in the microbial population count of six freshly made paint samples (PS1 - PS6) were monitored fortnightly for a period of 10 months. The growth data of isolated organisms from the fresh and spoilt paint samples were fitted into a multiple linear regression model to predict shelf life for the fresh paint samples. The microbial population ranged from 1.0×10^1 - 4.7×10^5 cfu/ml and from 1.0×10^1 - 5.5×10^3 cfu/ml for bacteria and fungi over the study period. Physico-chemical parameters such as specific gravity (SG), optical density (OD), transmittance (TR), pH and viscosity (VIS) were also determined every two weeks for the fresh paint samples over the ten-month study period. The measurements of the physico-chemical parameters suggested deterioration related to microbial population count of the paint samples. Consequently, the model developed comprised of two equations with particular attention to microbial population count and physico-chemical parameters of the paint samples. The microbial population counts of the spoilt paint samples were 3.4×10^{10} cfu/ml and 3.2×10^5 cfu/ml for bacteria and fungi respectively. The changes in the physico-chemical parameters ranged from 2.8658 - 1.0853, 1.49 - 3.91, 6.9 - 2.3, 8.5 - 5.6, 11.7cst -10.8cst for SG, OD, TR, pH and VIS in fresh paint samples. The percentage residual error between the shelf life predicted and the shelf life experimental ranged between 0.001 and 0.500. The shelf lives obtained for the fresh paint samples were 19, 21, 23, 22, 37, and 22 months respectively.

INTRODUCTION

Paint is the general term for a family of products applied to various surfaces such as wood, metal or stone to protect the surface from corrosion, oxidation and environmental weathering. It also provides decorative finish [1]. Water-based paints are prone to biodegradation because of their aqueous nature and presence of microbial nutrients. Therefore, the monitoring of paint quality is one of the main goals of the paint industry. The shelf life of water-based paints is influenced by a number of factors such as initial microbiological quality of raw materials, packaging materials and the manufacturing plant itself [4]. The paucity of information on paint shelf life is a major problem bewildering the paint industry. This is the reason for the extensive use of lead to improve paint durability and hence, shelf life [9]. However, the use of lead in paints has posed significant health hazard since the mid 1920's [5,9]. The aims of the present study were to determine the shelf lives of paints using a multiple linear regression model and to evaluate the validity of the developed model in predicting shelf life by comparing the observed result with the model prediction using the residual error factor [12].

MATERIALS AND METHODS

Microbiological Enumeration and Identification

Six paint samples (PS1-PS6) from a major paint industry in Lagos, Nigeria were collected randomly shortly after completion of production. They were monitored for microbial growth using standard plate count technique. Aliquots (0.1 ml) from both low (10^{-2}, 10^{-4}) and high (10^{-6}, 10^{-8}) ten -fold serial dilutions of paint samples were plated out on Nutrient agar (NA), Mac Conkey agar (MCA) and Potato dextrose agar (PDA) plates in three replicates. NA and MCA plates were incubated aerobically at 37^{0}C for 1-2 days and PDA plates at room temperature ($30\pm2_0$C) for 3-5 days. Precautionary measures such as aseptic techniques were adhered to and found effective as the control sample which was not opened regularly for isolation had same microbial population density as the experimental samples. In addition, spoilt paint samples were analyzed to know the level of microbial contaminants that could cause spoilage of water- based paint samples. The colonies that developed in the plates were enumerated. Pure cultures of isolates were obtained by subculturing and identified by reference to API (Analytical Profile Index) test systems.

Determination of Physico-chemical Parameters

Specific gravity: Specific gravity was determined by pycnometry as described by Ohwoavworhua and Adelakun [8]. A sterile pycnometer of 50 ml capacity was weighed and the weight recorded as M1. Paint sample (50 ml) was transferred into the pycnometer. The pycnometer and its content were weighed and the weight recorded as M2. The pycnometer containing the paint sample was filled with distilled water and shaken many times to allow all trapped air within the pycnometer to be expelled. The weight of the pycnometer was recorded as M3. The pycnometer was emptied, washed and refilled with distilled water and the weight recorded as M4. Specific gravity was then calculated using the formula:

$$SG = \frac{(M2-M1)}{(M4-M1)(M3-M2)}$$

Optical Density and Transmittance: This was determined colorimetrically [11] with a photoelectric colorimeter (Model: AE-11C Tokyo Erma Optical works Ltd, Japan). The colorimeter was standardized by adjusting it to read 100% light transmittance with 5 ml distilled water at a wavelength of 660 nm. The colorimeter had two scales. The bottom scale displayed the absorbance while the top scale, % transmittance. Five (5 ml) of serially diluted paint samples were poured in the cuvette and subsequently placed in the colorimeter to determine the optical density and the transmittance.

Mean pH: The pH of the paint samples was determined with a pH meter (Model: Jenway M50/Rev CE350EU) in 1: 200 solution of the paint samples in distilled water. The pH meter was calibrated using phthalate buffer (pH, 4.0) and phosphate buffer solutions (pH, 7.0).

Viscosity: Viscosity was determined using a glass capillary tubular viscometer (Model: Capirograph Toyoseikl Seisaku-Sho Ltd) as described by Rammohan and Yassen [10]. The paint sample was allowed to flow through an outlet tube (measuring tube which is narrowed into a capillary tube above the outlet). Two annular reference marks on the measuring tubes were used. The time it took the sample meniscus to drop from the upper to the lower reference mark was measured manually with a stop-watch. The viscosity was then calculated by multiplying the measured time by the viscometer calibration factor at room temperature (30 ± 2^0C).

Data Analysis

The microbial growth data for the fresh and spoilt paint samples were fitted into the first equation of a multiple linear regression model [12]. The regression parameter estimates ($ß_0$ - $ß_3$) were generated with data from the initial to the final microbial population count of fresh paint samples during the study period using the JMPIN software.

$$Y_i = ß_0 + ß_1 (\log X_1) + ß_2 (\log X_2) + ß_3 (\log X_3) + e \quad (1)$$

Where $ß_0$ is the regression intercept, $ß_1$- $ß_3$ are regression parameter estimates (at 0 -

10 months), X_1- X_3 (represented by the highest value of total bacterial count, total coliform count and total fungal count respectively) are the regressor coefficients at the spoilt state, Y_i is the time (in months) and e is the error factor. The data for the physico-chemical parameters during the study period were fitted into the second equation of the model:

$$Y_i = ß_0 + ß_1 (X_1) + ß_2(X_2) + ß_3(X_3) + ß_4 \ln(X_4) + ß_5(X_5) + e \quad (2)$$

Where $ß_0$ is the regression intercept, $ß_1$- $ß_5$ are regression parameter estimates (at 0 – 10 months), X_1- X_3 (represented by SG, OD, TR, pH and VIS) are the regressor coefficients at the spoilt state, Y_i is the time (in months).

Comparison between Observed and Predicted Shelf Life

This was based on the residual error factor ($e = Y_i - \hat{Y}$.) The observed microbial population count and physico-chemical parameters were compared with the predicted values.

$$e = Y_i - \hat{Y}$$

Where Y_i is observed result, $\hat{Y}$ is predicted result and e is the error factor

RESULTS AND DISCUSSION

Ten morphologically different microorganisms were isolated following initial ten-fold serial dilution and standard plate count from fresh paint samples (PS1- PS6) that have been processed to meet company's regulations and specifications. The bacterial isolates which were identified by the identification profiles generated using the database code obtained from the API identification software (APIWEB) include the following: *Bacillus polymyxa*, (OB-1) *Bacillus brevis*, (OB-2) *Bacillus laterosporus*,(OB-3) *Proteus mirabilis*, (OB-4) *Escherichia coli*, (OB-5) *Lactobacillus gasseri* (OB-7) and *Lactobacillus brevis* (OB-8) The fungal isolates were identified as *Aspergillus niger*, (OB-9) *A. flavus* (OB-10) and *Penicillium citrinum* (OB-11) respectively based on macroscopic and microscopic characteristics. The microbial population count data from the six paint samples (PS1- PS6) during the ten-month study period showed an increase from 1.0×10^1 to 4.7×10^5 cfu/ml. The mean changes in microbial population density of the paint samples are shown in Fig. 1. There was a protracted lag period of 4 – 5 months in the paint samples. Subsequently, there was steady exponential growth till the 10th month. The high counts of bacteria observed in the fresh paints (Fig.1) suggests that the shelf life of the paints would be rather short. The results obtained in this study demonstrate that microorganisms utilized the paints as a source of nutrients and that the constituents of paints were conducive to increased cell multiplication and population buildups in the paint. These were similar to the observations made by Gillatt [4] that a can of water-based paint is highly susceptible to deterioration. Similar observation by Da Silva [2] also proved that the various organic constituents of paints such as pigments, additives, binders etc. act as nutrients for microorganisms and help to stimulate microbial growth. In addition to these organisms, the spoilt samples were observed to contain *Pseudomonas aeruginosa* (OB-6). The microbial population densities of the spoilt paint samples are presented in Table 1. Previous investigators have shown that *Pseudomonas aeruginosa* is a commonly encountered organism in spoilt paints [7] and constitutes at least 75% of isolates from spoilt emulsion paints [3]. Spoilt emulsion paints have become a source of concern to marketers and consumers and now constitute a major problem bewildering the paint industry in Nigeria. The mean changes in physico-chemical parameters in the six paint samples are illustrated in Figs. 2 & 3. There were increases in optical density over the period for all the samples ranging from 1.49- 3.91. In contrast, there was a decreasing trend in other parameters ranging from 2.8658 – 1.0853, 5.2 – 2.3, 8.4 – 5.6 and 11.7cst – 10.8cst for SG, TR, pH and VIS respectively during the period. The physico-chemical parameters of the spoilt paint samples were 0.1058, 7.51, 0.15, 4.12 and 3.0cst for SG, OD, TR, pH and viscosity respectively. The changes observed for these parameters emphasized the increase in microbial growth and hence increase in acidic metabolites and lower viscosity. The physico-chemical parameters measured in the study appear to provide a sensitive and reliable measure of paint deterioration. Furthermore, the changes in the physical appearance of the paint samples over the study period showed a steady and gradual loss of the original colour, texture and viscosity. In view of the consistent increase in microbial population counts and the corresponding steady decline in physico-chemical parameters, the model used in this study was based on changes in microbial population count and physico-chemical parameters. Shelf life determination must therefore, depend on such criteria which include the rate of deterioration of quality parameters. Due to the fact that the stability of these physico-chemical parameters infer quality, their stability was regarded as a key factor or determinant of shelf life of paints. The data from the microbial population count and the physico-chemical parameters of fresh paints monitored during the period were combined with the corresponding data of the spoilt paint and fitted into the developed model to extrapolate the shelf life of paints. The shelf lives of the paints were therefore estimated to be 19, 21, 23, 22, 37, and 22 months respectively. The percentage residual error calculated by deducting the predicted results from the observed results was found to be marginal, ranging from 0.001 to 0.5 %.

The regression (ANOVA) was highly significant at a P value of 0.0001 indicating a good fit, which implies that the regression is very useful. Therefore, changes in physico-chemical parameters and microbial population counts can be used as indices to predict shelf life of products such as paints.

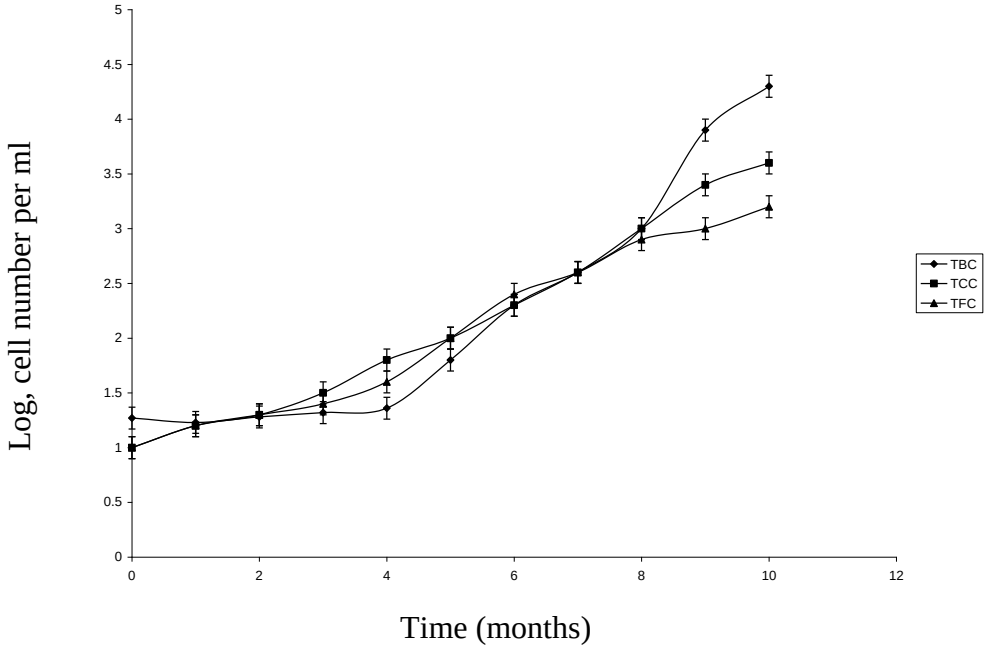

Figure 1: Mean changes in microbial population density in fresh paint samples PS1-PS6. TBC, total bacterial count; TCC, total coliform count; TFC, total fungal count. Data represent the averages of triplicate determinations.

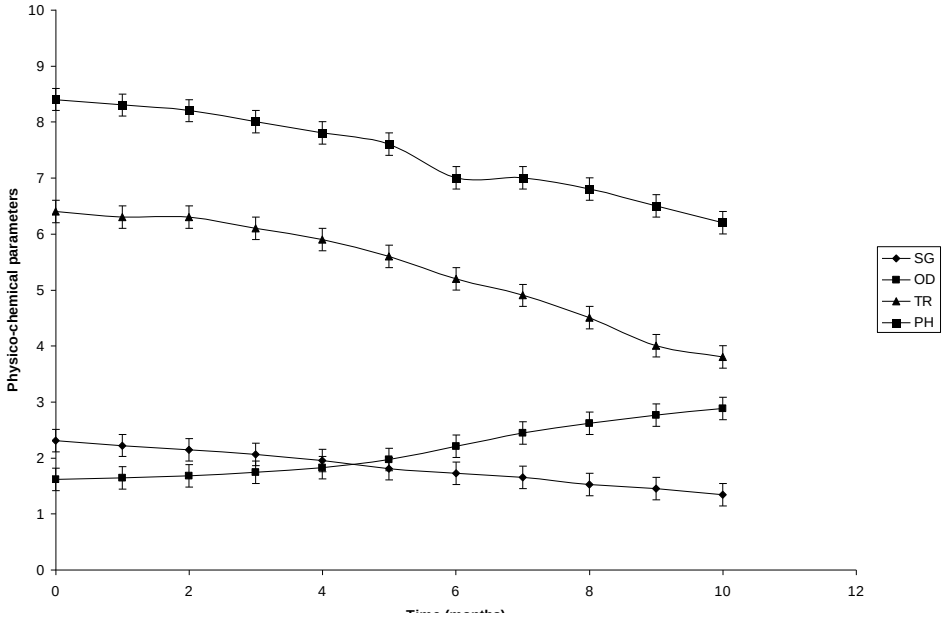

Figure 2: Mean changes in the physico-chemical parameters in fresh paint samples PS1 - PS6. SG, specific gravity; OD, $_{600\ nm}$; TR, transmittance. Data represent the averages of triplicate determinations.

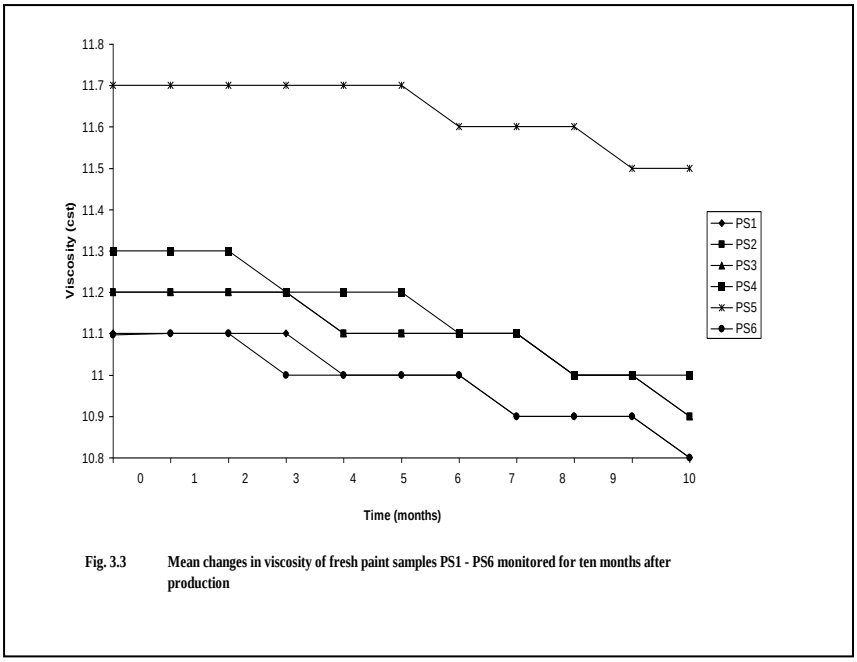

Fig. 3.3 Mean changes in viscosity of fresh paint samples PS1 - PS6 monitored for ten months after production

Figure 3: Mean changes in viscosity of fresh paint samples PS1–PS6 monitored for ten months after production.

Table 1. Microbial Population Densities in Spoilt Paint Samples

Paint sample	Total bacterial counts (x 10^{10} cfu/ml)	Total coliform counts (x 10^{7} cfu/ml)	Total fungal counts (x 10^{5} cfu/ml)	Fungal isolates	Bacterial isolates
PSA	2.9	1.1	2.5	OB-9	OB-2, OB-3, OB-4, OB-6, OB-7
PSB	3.4	1.1	3.2	OB-9, OB-11	OB-1 OB-6, OB-7, OB-8
PSC	3.0	1.0	2.8	OB-10, OB-11	OB-3, OB-4, OB-6, OB-7
PSD	2.5	2.9	2.5	OB-10	OB-2, OB-4, OB-6
PSE	3.1	1.1	2.2	OB-11	OB-1, OB-5, OB-6

Values presented are means of triplicate samples.

CONCLUSION

The microbial population count appeared to correlate with the degree of acceptability as indicated by changes in physico-chemical parameters of the paints. The model developed in this study was based on the microbial growth data and the physico-chemical parameters. On the basis of the results above, the microbial population count equation of the regression model predicted the paints shelf life to have an average nominal shelf life of 21 months, while the physico-chemical parameter equation predicted an average nominal shelf life of 24 months. The developed model can be adopted by the paint industry to make a reliable prediction of paint shelf life. The adequacy and accuracy of the developed model determined by the correlation coefficient which was greater than 0.90 indicate a good fit.

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