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Quantitative analysis of some brands of chloroquine tablets marketed in Maiduguri using spectrophotometric and high performance liquid chromatographic methods

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Abstract
The experiment involves analysis of chloroquine using ultra violet spectrophotometer in the ultraviolet range (200-400nm) and high performance liquid chromatography(HPLC) in which the samples were dissolved in various solvents and their various absorbance, peak area at various wavelength were determined and compared with that of the standard, wavelength of maximum absorbance at 331nm for chloroquine. Percentage and milligram content for each sample was determined so as to note if it was within the acceptable range of (92.5-107.5%) for chloroquine. For those that passed the test or if it was below or above the range for samples that are substandard or highly concentrated. The samples' absorbance and peak area was used along side with the standard absorbance and peak area to calculate the percentage content of each sample. It was observed that of the six samples of chloroquine tablet, none passed the test using UV spectrophotometer while Dana chloroquine with 98.7%, Evans chloroquine 107.3% and Palquine 106.4% passed using HPLC.

Keywords: Chloroquine Tablet, UV, HPLC

INTRODUCTION

There has been an overwhelming rise in generic drug products in the pharmaceutical market today. The introduction of generic drug products from multiple sources into health care delivery system of developing countries was aimed at improving the overall health care delivery system in such countries. However, this has been accompanied by a variety of problems of which the most critical is the widespread distribution of fake and substandard drug products. (Adebolagun *et al.*, 2007).

In view of this, therapeutic equivalence studies are carried out, whereby evaluative studies on biopharmaceutical equivalence, bioequivalence, bioavailability (for determination of drug approvals into markets) and chemical equivalence of generic drug products in the market are analyzed in comparison to the specified standard or branded products. The first stage in ascertaining the therapeutic equivalence of

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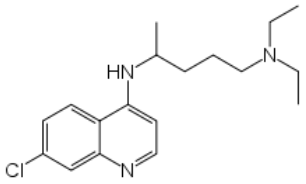
any drug product involves ascertaining the chemical and biopharmaceutical equivalence of such drug product. In conducting these studies, pharmaceutical analytical processes are used. Pharmaceutical analyses that are carried out are based on those specified by official books such as B.P, N.P, or from new research methods developed by scientist which are simple, inexpensive and rapid for evaluating the quantity and quality of drugs.

Chloroquine is one of a largest series of 4-amino quinolines synthesized as part of the extensive cooperative program of antimalarial research in the U.S during World War II. (Goodman and Gilman, 2008). Chloroquine was discovered in 1934 by Hans Andersag and coworkers at the Bayer laboratories, who named it "Resochin". It was introduced into clinical practice in 1947 for the prophylactic treatment of malaria (Krafts *et al.*, 2012). The *d*-, *l*- and di- forms of chloroquine have equal potency against *P. lophurae* malaria, but the *d*-isomer is somewhat less toxic than the *l*-isomer in mammals.



Chloroquine ($C_{18}H_{26}ClN_3$).
Systematic name: (*RS*)-*N'*-(7-chloroquinolin-4-yl)-*N*,*N*-diethyl-pentane-1,4-diamine

At the doses used for prevention of malaria, side effects include gastrointestinal problems, stomach ache, itch, headache, postural hypotension, nightmares and blurred vision. Chloroquine-induced itching is very common among black Africans (70%), but much less common in other races. It increases with age, and is so severe as to stop compliance with drug therapy. It is increased during malaria fever; its severity is correlated to the malaria parasite load in blood. Some evidence indicates it has a genetic basis and is related to chloroquine interaction with opiate receptors centrally or peripherally (Ajayi 2000). A metabolite of chloroquine, hydroxychloroquine, has a long half-life (32-56 days) in blood and a large volume of distribution (580-815 L/kg). The therapeutic, toxic and lethal ranges are usually considered to be 0.03 to 15 mg/l, 3.0 to 26 mg/l and 20 to 104 mg/l, respectively. However, nontoxic cases have been reported in the range 0.3 to 39 mg/l, suggesting individual tolerance to this agent may be more variable than previously recognized (Molina, 2011).

EXPERIMENTAL

Different brands of chloroquine, were used for the study. Pure sample of the drugs were obtained from NAFDAC which served as standard. The methods employed are the UV visible Spectrophotometric and high performance liquid chromatographic methods (BP 2008). The tablets were assayed spectrophotometrically using the following procedures. The average weight of the tablets from each sample was determined by weighing ten (10) tablets. The equivalent weight of 50mg of each brand was used. 0.1N HCl solution as blank. The absorbance of each sample was determined at the wavelength of 331nm. The same procedure was repeated using 50mg of the powdered standard and the absorbance determined and from which the % content and mg content was determined as follows:

$$\% \text{ content} = \frac{\text{Absorbance of sample} \times 100}{\text{Absorbance of standard}}$$

$$\text{mg content} = \frac{\% \text{ content} \times \text{Manufactures claim}}{100}$$

(Sani *et.al.*, 2012a)

The HPLC method (BP 2008) was followed. HPLC conditions included: Column: silica; Wavelength: 320nm; Flow rate: 1ml/min; Mobile phase: methanol/water (30:70). (Sani *et al*., 2012b)

RESULTS

Tables 1 and 2 show the result of UV spectrophotometric analysis used to calculate the percentage content and milligram content

of the drugs. The following calculation shows the results from the HPLC method in analysis.

$$\% \text{ content} = \frac{\text{Peak area of sample}}{\text{Peak area of standard}} \times 100$$

$$\text{mg content} = \frac{\% \text{ content}}{100} \times \text{Standard claim}$$

Table 1: UV absorbance of chloroquine at 331nm (E1%)

Sample	Absorbance
Maxquin	149.88
Danaquin	296.41
Palquine	1588.2
Evans	248.30
Quinox	222.01
Sa'aquin	392.76

Table 2: Percentage content and milligram content of different brands of chloroquine using UV

Sample	% content	mg content
Maxquin	3.86	5.8
Danaquin	7.6	11.4
Palquine	40.9	61.4
Evans	6.39	9.6
Quinox	5.7	8.6
Sa'aquin	10.1	15.15

Sample ID: CHLOROQUINE STD 2 200313 Vial: 200 Injection Volume: 20

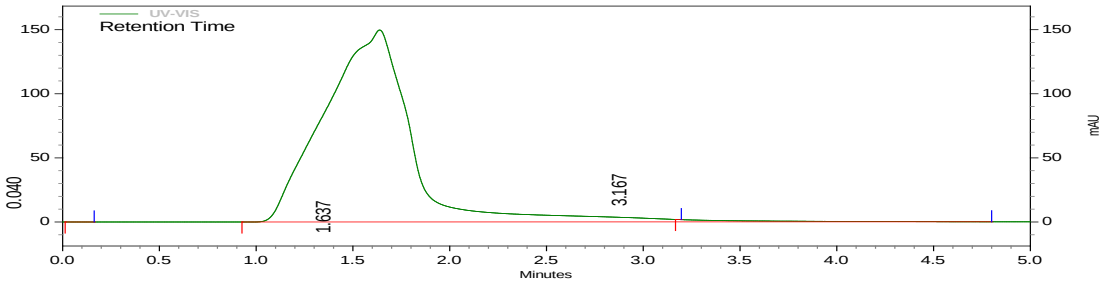


Figure 1: HPLC chromatogram of chloroquine standard

UV-VIS Results

Name	Retention Time	Area	Area Percent	Integration Codes
	0.040	348	0.002	MM
	1.637	18742308	99.998	BB
	3.167	14	0.000	MM
Totals		18742670	100.000	

Sample ID: DANA QUIMAL 210313 Vial: 200 Injection Volume: 20

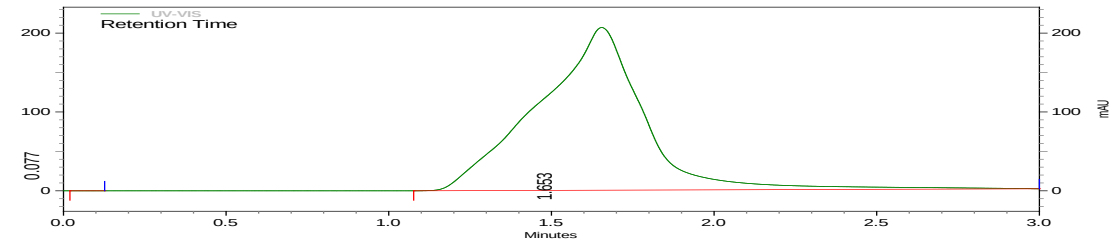


Figure 2: HPLC chromatogram of chloroquine (Dana)

UV-VIS Results

Name	Retention Time	Area	Area Percent	Integration Codes
	0.077	293	0.002	MM
	1.653	18489685	99.998	BE
Totals		18489978	100.000	

Sample ID: EVANS CHLOROQUINE PHOSPHATE 200313 Vial: 200 Injection Volume: 20

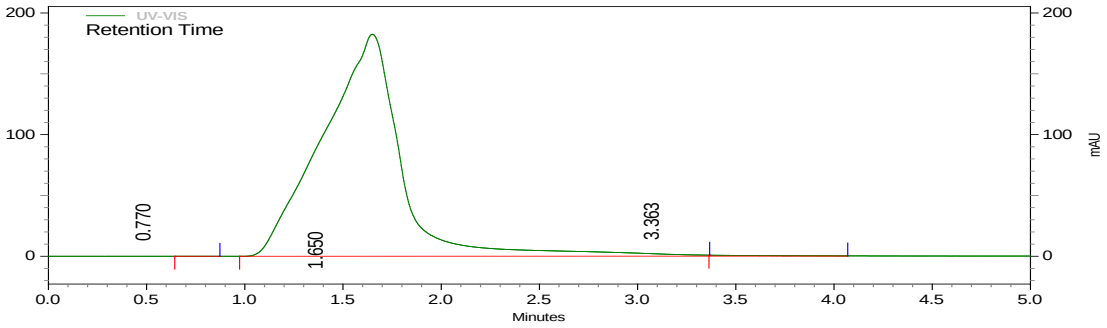


Figure 3: HPLC chromatogram of chloroquine (Evans)

UV-VIS Results

Name	Retention Time	Area	Area Percent	Integration Codes
	0.770	976	0.005	MM
	1.650	20103460	99.995	BB
	3.363	0	0.000	MM
Totals		20104436	100.000	

Sample ID: QUINNOX 200313 Vial: 200 Injection Volume: 20

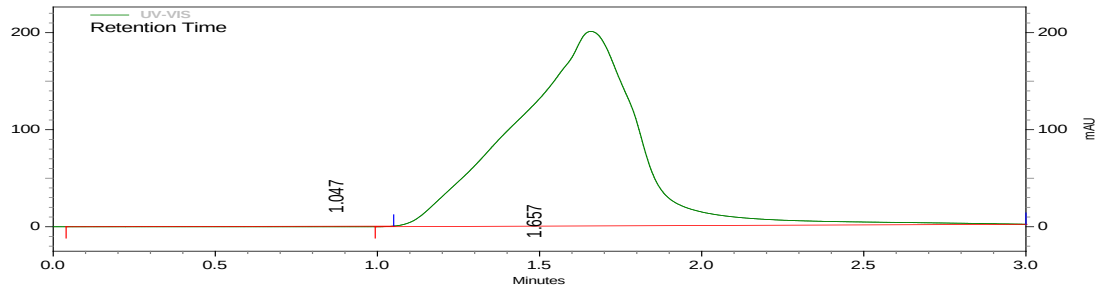


Figure 4: HPLC chromatogram of chloroquine (Quinox)

UV-VIS Results

Name	**Retention Time**	**Area**	**Area Percent**	**Integration Codes**
	1.047	79124	0.380	MM
	1.657	20768563	99.620	BE
Totals		20847687	100.000	

Sample ID: MAXQUINE 210313 **Vial: 200** **Injection Volume: 20**

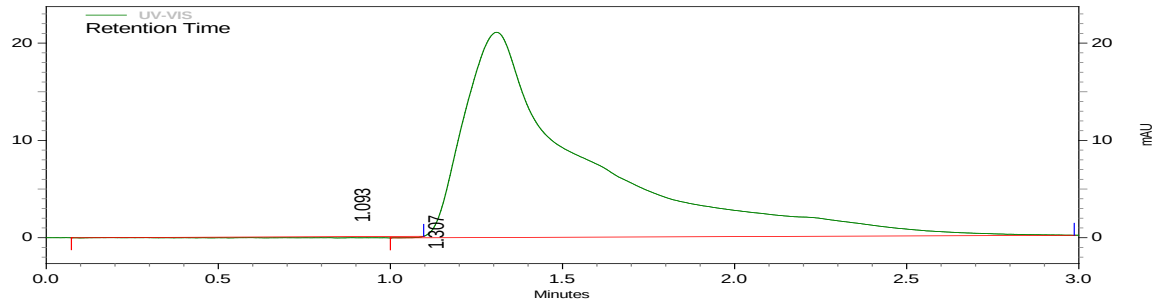


Figure 5: HPLC chromatogram of chloroquine (Maxquine)

UV-VIS Results

Name	Retention Time	Area	Area Percent	Integration Codes
	1.093	17961	0.875	MM
	1.307	2033563	99.125	BE
Totals		2051524	100.000	

Sample ID: PALQUINE 210313 **Vial: 200** **Injection Volume: 20**

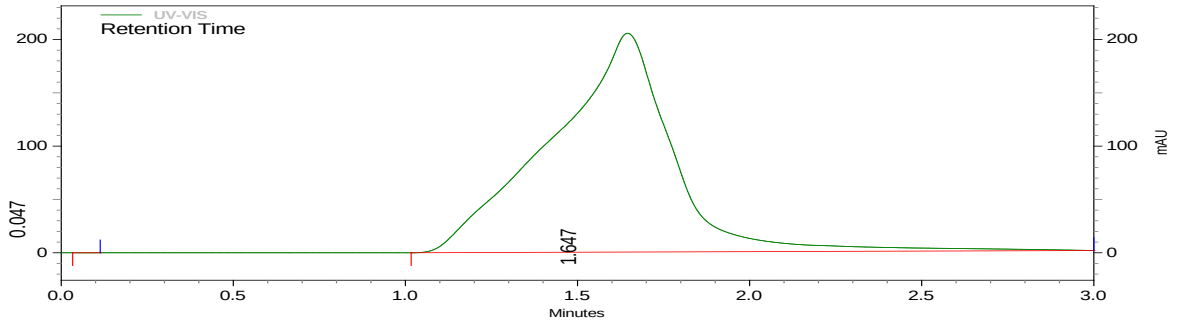


Figure 6: HPLC chromatogram of chloroquine (Palquine)

UV-VIS Results

Name	Retention Time	Area	Area Percent	Integration Codes
	0.047	310	0.002	MM
	1.647	19934336	99.998	BE
Totals		19934646	100.000	

Sample ID: SA'AQUINE 210313 **Vial: 200** **Injection Volume: 20**

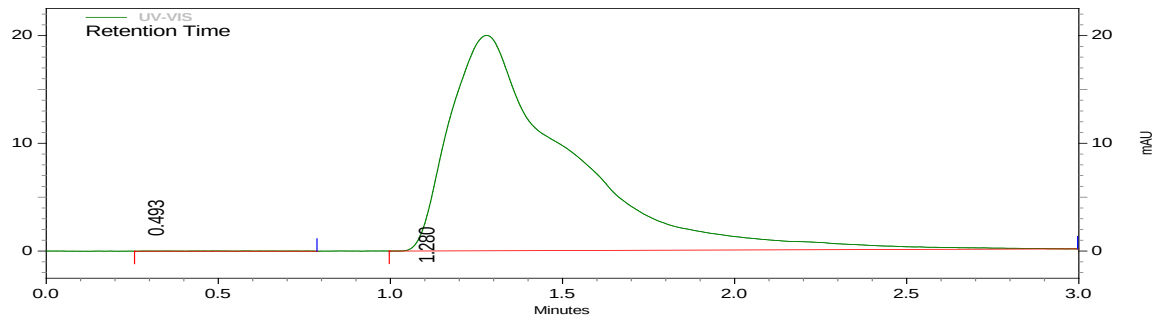


Figure 7: HPLC chromatogram of chloroquine (Sa'aquine)

UV-VIS Results

Name	Retention Time	Area	Area Percent	Integration Codes
	0.493	1963	0.103	MM
	1.280	1900525	99.897	BE
Totals		1902488	100.000	

Table 3 Percentage and milligram content of different brands of chloroquine using HPLC

Sample	% content	mg content
Maxquin	10.9	16.3
Danaquin	98.7	148.1
Palquine	106.4	159.6
Evans	107.3	160.9
Quinox	110.8	166.2
Sa'aquin	10.1	15.2

DISCUSSION

As stated by the British Pharmacopoeia, a Chloroquine tablet should contain not less than 92.5% and not more than 107.5% of the stated amount of chloroquine (BP 2008). A standard chloroquine tablet has an absorbance of 3887.5 at a wavelength of 331nm. From the result obtained using UV visible spectrophotometer Maxquin with percentage content of 3.86%, Danaquin 7.6%, Palquin 40.9%, Evans chloroquine 6.39%, Quinox 5.7% and Sa'aquin 10.1% is said to have failed the test as it is below the BP specified limit.(BP 2008).

For chloroquine tablet, Dana chloroquine with percentage content of 98.7%, Evans chloroquine 107.3% and Palquine 106.4% are said to have passed the test because they are within the set limit by the BP while Quinox with percentage content of 110.8%, Maxquine 10.9% and Sa'aquine 10.1% are said to have failed the test because they are either below or above the set limit by the BP (B.P 2008).

The result obtained from the U.V spectrophotometer is different from the result obtained in the HPLC and this variation could be attributed to the following causes of poor quality of drug products,

Conclusion

Following the BP specification, it can be concluded that Maxquine, Danaquine, Palquine, Evans, Quinox and Saaquine failed the test using UV but using HPLC Danaquine, Evans and Palquine passed the test while Quinox, Maxquine and Saaquine failed the test.

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